

INTERNATIONAL Chemical Engineering and Process Industries

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Topics of the Month

Fertiliser economics

THE prices of most fertilisers in Britain have almost doubled in little more than 12 months. Some fertilisers may not quite have risen by 75% in price; others may have risen more. At a time when the prices of most materials are rising steeply, fertilisers could not be expected to remain unaffected. Unfortunately, recent inflationary tendencies have coincided with the Government's decision to remove the 1940-50 subsidies and in one year British farmers have had to face a sharp swing from artificial cheapness to current high-cost reality. No one in the industry has much hope that the expansion in fertiliser use of the past decade can continue. The optimists would seem to be those who believe that the present rate of use can be maintained. In any case the shortage of sulphuric acid, with its particular bearing upon superphosphate production, stands in the way of further expansion.

It is not generally realised that for very many years fertilisers have been unrealistically cheap. Between the wars much of the industry was based upon by-products. Superphosphate in Western Europe became a 'vehicle' for the disposal of surplus sulphuric acid and there was considerable price-cutting and dumping. Basic slag has always been a by-product from the steel industry. The wartime decision to subsidise fertilisers so that their prices

remained at 1939 or 1940 levels perpetuated much of this artificial economic situation. Thus, where consumers of other materials must today face price increases that reflect the economic changes of 1949-51, fertiliser users are facing price increases that virtually reflect the change from the 'thirties to the 'fifties. At the same time the return farmers can obtain for their own products is determined by negotiation with the Government and the controlled maximum prices arrived at do not fully compensate for rises in costs of production.

The major influence determining the use of fertilisers is the simple answer to a quite simple question, 'Does it pay?' And in fact this becomes in practice a subtly different question, 'Does the farmer who has to buy the fertiliser believe that it pays?' The danger ahead is that the margin between profit and loss in the use of fertilisers may be too slender. If so, many farmers will not be confident enough to invest much money in fertilisers; and others, who have sufficient scientific knowledge, may feel that the other influences that affect the final crop yield, notably weather, are too uncertain to justify the maximum investment in fertiliser applications. It is a major duty of the British fertiliser industry to devote far more attention to sound technical publicity than it has previously considered necessary.

Industrial floor finishes

THE floor is the least likely part of a building to attract much attention, either from the people who occupy it or from the casual visitor. This is particularly the case with industrial buildings in spite of the fact that the selection of suitable finishes is of concern to the architect, the builder, the management, the safety and welfare officer and the operatives. The importance of floors is now emphasised by the appearance of a booklet, 'Floor Finishes for Industrial Buildings,' by G. E. Bessey, of the Building Research Station, which has been published by the Stationery Office. Until we read this booklet we did not appreciate the surprising diversity of flooring materials available for factories. No fewer than 21 are discussed in this practical booklet, besides 9 types of bedding and jointing materials for floors.

To quote the booklet, 'The main features that govern the choice of an industrial floor finish are durability under the particular conditions of use, comfort and convenience to the user, and appearance . . . The most important of these is, undoubtedly, durability . . . Comfort, with which may be included safety, is also important . . . Appearance is less important for industrial floors.'

Most flooring materials are susceptible to some form of chemical attack. The agents which may cause it are not only industrial chemicals but include such seemingly innocuous substances as milk, sugar and fruit juices, all of which affect Portland cement concrete, and animal and vegetable oils and fats, which affect both concrete and asphalt. Prevention of spillage, frequent washing and the provision of adequate falls for drainage are all of great help in minimising chemical deterioration.

The effect of floor finishes upon the incidence of accidents in a factory may be considerable. Hazards include slipperiness, uneven wear, pot-holing, indentation or splintering of surfaces, cracking or lifting of the floor, bad design of steps or ramps, and the use of materials which may lead to fire and explosion. Indirect effects upon safety include risks arising from the greater fatigue induced by walking on some floors than on others.

Most heavy duty industrial floors fall in one of these groups: concrete including special granolithic type floors and precast concrete tiles; mastic asphalt or pitch mastic jointless finishes; clay tile finishes; timber finishes; metal finishes; and stone floors. Flooring for chemical factories raises special problems which are beyond the scope of the booklet. Nevertheless, problems of certain process industries are considered in detail and recommendations made. For instance, for factories processing meat or animal or vegetable fats and oils, dense clay flooring tiles, jointed in high alumina cement, are recommended. For factories using processes involving sugar solutions and weak acids, it is stated that the use of precast concrete blocks will facilitate repairs and that high alumina cement is likely to reduce deterioration. Hard, acid-resistant mastic asphalts and acid-resistant pitch mastics are also recommended for certain conditions. High alumina cement concrete, rubber or hardwood finishes are suggested for factories using salts or salt solutions, while hard, dense ceramic tiles or a good quality granolithic concrete are advised for breweries. Floors exposed to strong acids are recommended to be finished with acid-resistant mastic asphalt or with paving bricks or tiles set in acid-resisting cement. For heavy engineering factories granolithic concrete is widely used and steel or cast iron plates, tiles or grids are often the most suitable finishes for areas subject to heavy abrasion.

Gas industry production

A BIG increase in the value of the output of the gas supply industry during the years 1935-48, is shown in the latest preliminary report (No. 151) of the Census of Production. This report gives comparative statistics for the years 1935 and 1948. In the former year, Great Britain's gas industry's output was worth £63,880,000; in the latter £181,557,000. These figures, of course, reflect the universal rise in prices during the years under review as do those for the cost of materials, fuel and electricity used—£100,795,000 against £25,096,000. Incidentally, these figures show that the gas industry, up till 1948, at least, pursued the commendable anti-inflationary course of not passing on all its increased costs to consumers. Increased efficiency probably accounts for the fact that while the cost of its raw materials rose more than fourfold between 1935-48, the industry was able to keep down the price of its products to less than three times 1935 levels. This supposition is partly borne out by the figures for net output per person employed. This rose from £328 to £613. It is interesting to note that the employees of gas companies increased their output more than those of local authority gas undertakings. The former's output rose from £314 to £640, the latter's from £364 to £552. Grand total of workers rose from 119,586 to 131,821.

The industry is, of course, a big buyer of plant and machinery; in 1948 it spent £10,418,000 on plant and machinery of all kinds out of a total capital expenditure of £18,611,000.

Exit oil seed pressing?

RECENT American reports suggest that solvent extraction processes are steadily displacing screw or hydraulic methods of pressing in vegetable oil extraction. In rather less than 10 years solvent extraction has developed from a pilot-scale experiment into a major technique in this old-established branch of chemical industry. For example, there are at least eight manufacturers of plant in the United States now marketing large continuous extraction systems; and already about 65% of the soya bean oil produced is obtained by solvent extraction, a proportion that will shortly rise to 75% with the completion of plants now being erected.

The principal advantage of solvent extraction is the higher recovery of oil. After pressing, the residue still retains some 5% of oil; after solvent treatment, only 1% is left. Other advantages are claimed, notably lower power and maintenance costs and less deterioration of the protein in the residual material. Under today's economic conditions, with vegetable oils commanding high prices and feeding-stuff protein becoming more and more important in cattle farming, the solvent extraction process produces its benefits at the most profitable points. The fact that extraction plants cost considerably more to instal than pressing plants has not discouraged their expansion.

The solvent currently favoured is trichlorethylene. Though dearer than hexane or heptane, it has been found to give a greater oil recovery and to provide a purer oil. Also, it is less inflammable than the cheaper hydrocarbon solvents originally used.

Nevertheless, the day of the press in this industry is not by any means ended. Vegetable materials that contain high percentages of oil, particularly tung nuts and ground nuts, have so far proved difficult to process by solvent extraction methods. These materials break up during extraction and the extractor tends to clog. The same

problem has been encountered with flax seed; it has been overcome by introducing a preliminary pressing treatment to remove some of the linseed oil, solvent extraction methods being used to deal with the residual cake. A similar partnership of pressing and solvent treatments is being used for extracting cotton-seed oil. It seems likely that for most oil-containing seeds or nuts the combined technique will eventually prove the most effective while solvent extraction by itself will become the exclusive oil-extracting method for a few particularly suitable materials like soya beans.

Mexico's first ammonium sulphate plant

LATIN America's first plant to produce ammonium sulphate has been opened at Cuautitlan, 20 miles from Mexico City, by President Aleman. Two officials of the U.S. Export-Import Bank also attended. Investment in the plant amounts to some \$10,000,000. Its capacity is 63,600 metric tons annually. Four million cubic feet of natural gas, and 65 metric tons of sulphur, the former piped in from the Poza Rica oilfields and the sulphur produced there with equipment which was installed late in 1950, will be used daily.

Owners of the new plant are Guanos y Fertilizantes de Mexico, a company in which the government's bond and investment corporation, Nacional Financiera, holds stock. The local investment was augmented by a credit extended to Nacional Financiera by the Export-Import Bank. In the opinion of Nacional Financiera advisers, production at the Cuautitlan plant will represent a seventh part of the nitrogen that is required to put Mexican farm lands back into capacity production. Total production of nitrogen in Mexico, estimated in terms of sulphate of ammonia, includes 2,800 metric tons annually at Nueva Rosita (Coahuila) and 10,000 tons derived from guano and other organic fertilisers. The nation's deficit when production at Cuautitlan is taken into account, is estimated at 423,600 metric tons annually. The value of imports of fertilisers in recent years were: 1947, 3,600,000 pesos; 1948, 1,100,000; and 1949, 3,600,000.

Nuclear power station project

ASTEP towards the use of atomic energy for the production of electric power has been taken in America where the Monsanto Chemical Co. and the Union Electric Co. last month signed an agreement with the Atomic Energy Commission for investigation of the possibilities of a nuclear power station. The agreement initiates a research project on the lines recommended by Monsanto's president, Dr. C. A. Thomas.

The proposal as outlined by Dr. Thomas a year ago consisted of three steps: first, a study to see if it is feasible to produce electric power as a by-product of a nuclear reactor; second, for a private concern to design and build such a plant; and, third, for the private company to operate it. Earlier this year the A.E.C. advised Monsanto it was ready to go ahead with the first step.

Dr. Thomas has outlined generally the method of operation which he believes the study will prove practical: The A.E.C. would send uranium to the private reactor to be used as a primary fuel. In the reactor the uranium would be transmuted to plutonium, which would be returned to the Government for its atomic energy programme. The high temperatures which would develop in the reactor during the transmutation process would be passed through a heat

exchanger where they would be converted to steam. The steam would be fed to normal-type turbines which, in turn, would operate generators that would produce electricity. It is hoped that the study will show that plutonium can be produced at lower costs than at the present.

Sulphur in the Andes

A MAJOR reason for the present world sulphur shortage is that over the years American sulphur has been so cheap that it put many former producers out of business and discouraged the development of deposits judged uneconomic by U.S. standards. Now that the situation has changed so drastically great efforts are being made to reactivate derelict workings and to develop formerly 'uneconomic' reserves. Hitherto most attention has been paid to the rehabilitation of the Italian sulphur industry, which is proceeding apace with strong American assistance. Now comes news of similar activity at the other end of the world. The country is Chile and the location of the sulphur ore, an estimated 20,000,000 tons containing 50 to 80% S, is the Andes mountains. So far, because of the inaccessibility of the deposits and the fact that some mine workings are at altitudes of no less than 20,000 ft., the Chilean sulphur reserves have only been nibbled at. In the six years to 1945, Chilean production hovered around the 30,000 ton p.a. mark, but since then it has declined to an annual rate of about 15,000 tons for the first six months of last year. Hard-pressed by the sulphur shortage, the big fertiliser firm of Fisons, whose new superphosphates factory we describe in this issue, sent a mission to Chile to investigate the chances of increasing output. This mission has studied the problems at first hand, but apparently has returned discouraged. Given the backing of a powerful organisation, undoubtedly Chilean production could be increased, but even in the present conditions of sulphur starvation the question of price cannot be ignored. In this context we are thinking not so much of American sulphur but of sulphur from pyrites, anhydrite and those other sources which are now being so actively developed. Is it too fantastic to suppose that in five years there may be a glut of sulphur on the world markets?

Big Co. plant reopened

AFTER ten years' idleness, due to shortage of foundry coke, the unique carbon dioxide plant of T. Wall & Sons, Ltd., an integral part of their North London ice-cream factory and a landmark on the Acton skyline for more than 20 years, has resumed production. The plant, German-built and first of its kind in Britain, produces dry-ice for ice-cream refrigeration. Five absorbing towers, 90 ft. high, have been refilled with coke and the entire plant has been overhauled by Mr. R. Northover, Wall's chief engineer, prior to refilling of the towers by Black (Installations), Ltd., of Ealing. Each tower holds about 80 tons of coke. Refilling was necessarily a long and tedious operation for which a skip-hoist crane was fixed to the top of the towers. These huge towers rely for stability upon their weight; there are no deep foundations or guy-ropes holding them erect.

It is expected that the renovated plant will go far towards fulfilling the dry-ice requirements of Wall's three factories—at London, Manchester and Edinburgh—and those of the numerous supply depots throughout England, Scotland, Wales and Northern Ireland.

A century of synthetic fibres

IT was not until shortly after 1851 that serious attempts were made to realise the old dream of making artificial silk. In 1857 Hughes obtained a patent for spinning a mixture of glue, starch, resins and tannins. Later, Swan produced threads by forcing through jets a solution of cellulose nitrate in glacial acetic acid, fabrics being made from such threads after denitration being shown at the London Exhibition of Inventions in 1885. It was Chardonnet, however, who put the process on a commercial basis, samples of Chardonnet silk being on view at the Paris Exhibition of 1889. Although this process is now obsolete, its development was of outstanding significance in the history of the chemistry of artificial textiles.

In 1891, Cross, Bevan and Beadle found that the treatment of cellulose with caustic acid gave a material which reacted with carbon disulphide to yield cellulose xanthate. Commercialisation of these reactions leading to the production of viscose rayon in this country was carried out largely under the influence of Courtaulds Ltd. In 1894 Cross and Bevan found that acetylation of cellulose occurred at atmospheric pressures in the presence of a dehydrating catalyst such as zinc chloride or sulphuric acid, the triacetate so obtained being soluble in chloroform. If hydrolysis of the triacetate is carried out so as to obtain a product halfway between di- and tri-acetate, this material is soluble in acetone. Following the use of cellulose acetate-acetone dopes for the coating of the fabric of aeroplane wings in the 1914-18 war, manufacture of cellulose acetate rayon was commenced in this country under the leadership of Dr. Henri Dreyfus. By 1921, *Celanese* was on the market.

More recently, regenerated protein fibres have resulted from an attempt to prepare artificial wool. *Lanital* and *Aralac* are made from casein and *Ardil* utilises the vegetable proteins of groundnuts. Proteins from the soya bean form the basis of a fibre developed by the Ford Motor Co. and used for motor-car upholstery. Further developments in the field of regenerated protein fibres can be expected.

Recalling these highlights of synthetic fibre development during the century since the great exhibition of 1851, Dr. J. L. Stoves, writing in our associate journal, *Textile Industries and Fibres*, states that a completely new field was opened with W. H. Carothers' work on high polymers, resulting in nylon, the first truly synthetic fibre. Since the pilot-scale production of nylon in 1938 many large plants have been built in the U.S.A. Without detracting from Carothers' work, the writer believes it true to say that his success only became possible as a result of a century's progress in chemical investigations and technical invention.

Other new fibres are *Terylene*, developed in Britain from terephthalic acid and ethylene glycol, while *Vinyon* is a co-polymer of vinyl chloride and vinyl acetate.

Vinylidene chloride, polyvinyl chloride, and acrylonitrile are also used for the manufacture of synthetic fibres.

The rising demand for nitrogen

DURING the past few months increased demands for nitrogen for agricultural and industrial purposes have been accompanied by a decrease in production, owing to the shortage of coal and sulphuric acid. The consequence is that so far this year world consumption has exceeded production by 150,000 tons. These facts are given in Aikman's half-yearly report on the nitrogen industry issued last month. The report states that if revised estimates of pro-

duction prove correct, supplies for 1951-52 and 1952-53 will only be sufficient for further increases in consumption of about 3% and 6½% respectively, so that if demand takes its normal course a rise in prices appears inevitable.

In Europe the coal shortage is tending to reduce production and it is expected that the shortage of sulphuric acid everywhere may result in a 20% decrease in the supply of sulphate of ammonia during the coming year, although a considerable portion of ammonia hitherto used in its production will go into other products. A big increase in American consumption for industrial and agricultural purposes, largely due to the Korean war, has turned that country from an exporter to an importer of nitrogen. Should the Korean war end, the demand for industrial purposes might lessen, but on the other hand, all indications point to large quantities being required for agricultural purposes in China, Korea and Formosa. Japan is understood to have exported during the current year about 28,000 tons of nitrogen to Formosa and 14,000 tons to Korea and there are indications that during the next few months further exports of about 20,000 tons may be made, probably to Far Eastern areas.

According to the report, negotiations between China and Russia for the export of a large quantity of nitrogen from Eastern Germany broke down and exports from there amounted to only 20,000 tons for the year against the expected availability of 50,000 to 60,000 tons. The reason is said to have been a bigger demand in Eastern Germany.

Owing to the rising price of coal and the shortage of sulphuric acid, costs of many synthetic nitrogen producers are believed to have risen during the year by 20 to 25%. In welcome contrast is the situation in Chile, where costs are thought to have risen only slightly, with the expectation of a sharp drop when the solar evaporation plants are completed and in production.

Improved solar still

A 'SOLAR STILL' to convert sea water into potable water was described as a commercial possibility and a solution to the problem of supplying water to arid countries by Dr. Maria Telkes of the Massachusetts Institute of Technology at a recent American chemical society meeting.

The solar still is built on the principle that distilled water can be obtained without heating the water to its boiling point. It consists of a large pan from which sea water is evaporated by means of the sun's rays penetrating a glass roof over the pan. The roof is kept cool enough by air and wind to condense the evaporated water, which runs down and is collected in gutters.

The solar still is not a new idea. In fact, a huge still covering nearly an acre of land was constructed in Chile in 1880. It supplied about 6,000 gallons of fresh water daily. Solar stills have not been developed further because of their inefficiency of operation as compared with their cost of construction.

Dr. Telkes' work has shown that the biggest obstacle to the efficient conversion of sea water has been the tremendous heat loss through the bottom of the pan and into the ground. The obvious answer to this problem—which has now been incorporated into the design of the new experimental models of the solar still—is heat insulation between the pan and the earth. According to Dr. Telkes it is probable that further development work will show that the insulated type of solar distiller can be built at an acceptable cost and that it can solve the problem of supplying water to arid lands.

A New Plant for Superphosphates and Compound Fertilisers

At Immingham Dock, on the Lincolnshire coast, Fisons Ltd. have put into production the largest fertiliser factory of its kind in Great Britain. The factory cost £4,500,000 and construction took four years. The buildings occupy 10 acres of the 45-acre site. The factory is the first in Britain to produce triple superphosphate fertiliser and is thus an important dollar-saving asset to the nation. Single superphosphate and granular compound fertilisers are also manufactured. The whole huge factory is mechanised and automatically controlled to a very high degree and only 350 people, including office workers, are needed to run it. Here is a detailed description of this unique undertaking.

ON April 27, 1951, the Parliamentary Secretary to the Board of Trade opened a new fertiliser factory built for Fisons Ltd. at Immingham, Lincolnshire. This is the largest plant of its kind in Great Britain and it will make a very substantial contribution towards making this country as independent as possible of imported supplies of superphosphate for agriculture. It will, therefore, save what has until now constituted a considerable dollar drain on the country's resources.

Fisons took the bold decision to construct this factory in response to an appeal by the Government to the fertiliser industry in 1944. It was a bold decision, because at that time nearly all the necessary materials which would be needed were in short supply and, in view of the heavy development programme for power stations, coal mining equipment and other schemes, this project could not be given high priority. This meant that throughout the construction period plans had to be altered from time to time to meet the ever-changing position with regard to scarcity of materials.

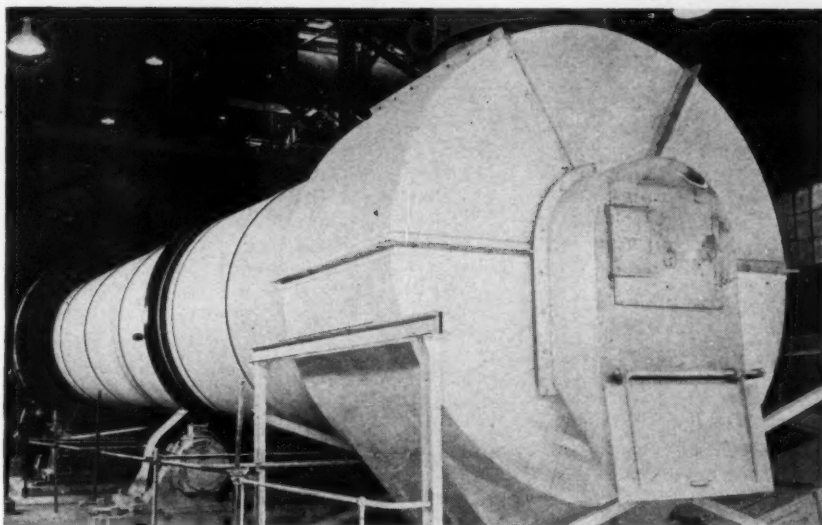
Fisons themselves, with their great experience of the fertiliser industry acquired over many years, evolved all the manufacturing process plant themselves, but appointed as joint consulting engineers to carry out the engineering side of the project Sir Alexander Gibb & Partners and Maunsell, Posford & Pavry.

Planning of the factory

The main planning, design and detailing of the various buildings, and the material handling, was apportioned to Maunsell, Posford & Pavry, and the design of the services was carried out by Sir Alexander Gibb & Partners.

The main factory site occupies an area of 45 acres adjacent to Immingham Dock on the Humber Estuary, which is one of the best deep-water docks on the east coast of England.

The factory was built here as it was found most economical to bring in the majority of the raw materials and also to export much of the finished product by sea. Full advantage was taken of this in planning the layout of the factory. About



Rotary drier in the triple superphosphate plant.

a mile away from the main site an area of 146 acres was acquired by Fisons for the construction, in stages, of a large reservoir to contain the gypsum which appears as a by-product in the manufacturing process.

The main materials to be imported annually into the factory are: sulphur 30,000 tons, phosphate rock 130,000 tons, sulphate of ammonia 30,000 tons, muriate of potash 10,000 tons, and coal 25,000 tons, all of which, with the exception of coal, are brought in by sea.

Exported from the factory annually are 70,000 tons of triple superphosphate fertiliser, 50,000 tons of single superphosphate fertiliser and 75,000 tons of granular compound fertiliser.

It was desired that the whole factory should be so planned as to use mechanisation to the greatest degree possible and, in point of fact, the whole of this large factory can be run with a total personnel of only 350, including office staff and shift workers.

The raw materials are unloaded from the ships' holds by two 5½-ton travelling grabbing cranes which discharge into two travelling hoppers at the quayside. These travelling hoppers are served with slow-

moving slat conveyors, which are so arranged that they discharge on to a 600-ft.-long conveyor belt installed in an elevated enclosed gallery running at the back of the quay. From these main incoming conveyor belts the raw materials are carried by other conveyors to the end of the factory most remote from the dock, where they are discharged into the various storage buildings. The flow of these raw materials then gradually works back throughout the factory, first into the basic process buildings and then on to the further process buildings, at which point they are joined by the salts, and so to the final product storage buildings. By this time the product has reached a point fairly close to the quayside again and a conveyor system then distributes the product to road, rail or sea transport.

There are 16 main buildings on the site and about 30 subsidiary buildings, together with 1½ miles of roadways and 2½ miles of rail tracks.

Having settled the main route for the flow of materials through the factory, the next problem was to arrange the juxtaposition of all these buildings so as to provide straight runs for the interconnect-

ing conveyor belts, and also to allow sufficient space for future extension of the factory. This was a difficult job, as the buildings had to be sufficiently far apart to enable conveyors which started their run on the floor of one building to finish up in the roof of another, while limiting the gradient of these conveyors to prevent the material from rolling back on itself. On the other hand, in the interests of economy, the buildings could not be too widely spaced. It was stipulated that elevators should be used as little as possible, otherwise this would, of course, be the obvious way of getting over the difficulty just mentioned.

Foundations

The first site problem with which the engineers had to contend was the determination of the type of foundations to employ. The ground is reclaimed land filled with spoil from the original excavation of the Immingham Dock, which was constructed early in this century. The top 6 to 9 ft. comprises silt and warp, which has little bearing capacity. From 9 to 30 ft. deep there is a blue silty warp, which also has very little bearing capacity, but which, in addition, has the unfortunate property of behaving as a fluid whose specific gravity is about two. This effect had to be very carefully taken into account in designing both temporary and permanent works in connection with deep pits and sumps, as the walls had to withstand this heavy fluid mud pressure and the base slabs had to be designed to withstand the induced upthrust. On account of the above-mentioned site conditions, it was only possible to construct very few buildings on a raft foundation and these were, of course, only small lightly loaded structures. All the rest of the structures on the site had to be supported on piles and, as little support could be obtained until a depth of about 30 ft. was reached, the engineers decided to use 'Franki' compressed *in situ* piles, of which 5,750 were driven.

The buildings fall into four main categories: process, storage, utility and administrative.

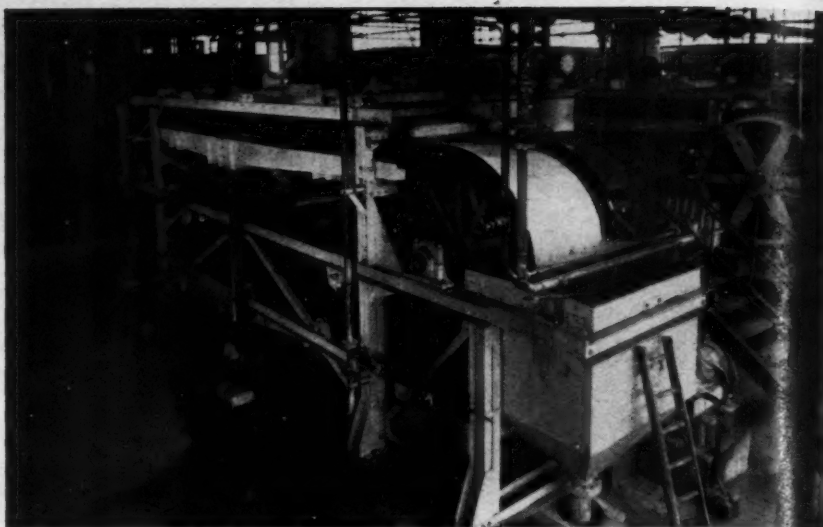
Process buildings

These buildings are those housing the sulphuric acid plant, the grinding plant, the single superphosphate plant, the phosphoric acid plant, the triple superphosphate plant and the batching and granulating plant.

Sulphuric acid

The sulphuric acid plant was designed by Simon-Carves and it is one of the largest single-unit contact plants in Europe.

It is of the latest design, burning sulphur and employing vanadium pentoxide catalyst in a four-pass converter. The waste heat is used to generate superheated steam which is fed to the power station, and makes a major contribution to the econo-



Landskrona filter in the phosphoric acid plant.

mics of the factory. The plant is rated to produce 250 tons/day of 100% H_2SO_4 with a conversion efficiency of 98%, and is the largest single-converter unit so far built in this country.

The plant comprises sulphur melting and settling pits, sulphur burner, hot gas filter, waste heat boiler, converter, economiser, atmospheric gas cooler, drying and absorber towers, acid cooling and dilution system, together with the necessary pumps and ancillaries, and a 440-h.p. blower. All instruments are centralised in a small control room, and the plant is operated by two men per shift.

Only a very limited amount of the plant is housed, the bulk of it being erected in the open, following modern American practice.

The product acid is stored at 94% strength in four large tanks having a total capacity of 6,000 tons of 100% acid.

Phosphate rock grinding plant

This is a steel-framed asbestos-clad structure of 50 ft. span and 63 ft. high to eaves. Due to the arrangement of the plant itself, no lateral support could be provided to the main stanchions, and they are, therefore, of very heavy section to withstand the wind loads on this exposed site. Vertical windows continuous for the maximum height, and patent glazing in the roof, provide ample lighting to all parts of the plant. A mess room, workshops and lavatory accommodation is provided in the building.

The plant itself comprises, among other things, some heavy mills which it was thought might engender vibration. This point had to be taken into account when designing the foundation for the plant and for the structure.

Four international combustion Lopulco roller mills are installed, each rated to grind 12 tons/hr. of rock phosphate to a fineness of 80% through a 100-mesh

sieve. Each mill is air-swept by its own fan and the product is recovered by cyclone. Adequate storage bins are provided both for unground and ground phosphate. The ground phosphate is conveyed to the various consuming plants by a Fuller-Kinyon pneumatic pump.

Single superphosphates

No very difficult engineering problems were met with in designing the building for the single superphosphate plant, where ample light and ventilation is provided around the mass of plant with its access ways and staircases.

For the manufacture of single superphosphate a Broadfield den has been installed with a rated capacity of 20 to 25 tons/hr. The evolved gases are vented to a scrubbing tower. The product is taken by conveyor belt to the superphosphate store.

Phosphoric acid

The building housing the phosphoric acid plant produced a number of problems. It is a steel-framed asbestos-clad building with horizontal continuous windows to give maximum light to the plant on the various floor levels. As the manufacture of triple superphosphate fertiliser is a new process in this country, the general arrangement of this process, the first part of which is the manufacture of phosphoric acid, was based on American practice. However, American plant was not used, and the engineers had first of all to provide for the carrying, in this multi-storey building, of the British manufactured plant, the weight and overall dimensions of which differed from its American counterpart, and also had to alter the plans so as to comply with British Standards of construction, which differ in a number of respects from American standards. The engineers also had to take care to observe the requirements of the Factory Inspector,

which requirements are, generally speaking, more stringent in this country than in the U.S.A. The upper floors of the building were constructed in reinforced concrete and all floors are arranged to drain to special channels, involving considerable complication in the reinforcement and the structural steel design.

The plant is built to the design of the Dorr Co. Inc. and operates the Dorr strong acid process. It is designed to treat 250 tons/day of ground rock, and comprises reaction, filtration and evaporation sections.

The reaction section, where the rock is treated with sulphuric acid in measured proportions, is in two halves, each having three pre-mixer tanks and four agitator tanks, in series. The liquor is recirculated, except for a proportion which is pumped to the filtration section. This contains five Landskrona belt filters by R. O. Stokes Ltd., for removal and washing of the precipitated gypsum, which is then reslurried with water and pumped to a large settling pond. The phosphoric acid from the filters has a strength of 32% P_2O_5 and this is concentrated to 38% P_2O_5 in the evaporation section, which comprises three cast-lead single-effect steam-heated evaporators by W. J. Fraser & Co. Storage tanks are provided for both 32% and 38% P_2O_5 strengths of acid. Four wash towers deal with the gases evolved by the reaction.

Triple superphosphate

The design of the seven-storey triple superphosphate plant posed problems similar to those of the superphosphate plant, but in this case the difficulties were enhanced by the nature of the plant. For instance, a rotary drier has to be accommodated in a fairly restricted space and the problem of the heat effect from this drier upon the structural steel had to be considered. This building involved some very heavy structural design to cater for



Sulphuric acid plant with daily capacity of 250 tons.

such things as pulverisers, vibrating screens, etc., situated on the upper floors.

This plant is also to the design of the Dorr Co. and has a capacity of 230 tons/day of triple superphosphate, which is produced in granular form. This requires the treatment of 100 tons/day of phosphate rock with phosphoric acid in lead-lined reaction tanks. A considerable proportion of recycled fine product is then added and the mass thoroughly kneaded in a large paddle-mixer known as a Blunger, two of which are installed. The mix is then dried in a rotary drier supplied by Ernest Newell Ltd., after which it passes to the screening section where the desired size fraction of granular product is removed. This is taken by conveyor belt to the triple superphosphate store. The oversize is crushed and recycled along with the undersize. The evolved gases are scrubbed in wash towers.

Granular compound fertilisers

A granulating plant is installed for the production of compound fertilisers, adjacent to both the superphosphate and salts store. The mixture is prepared by mixing weighed quantities of the con-

stituents, and wetting them to the right consistency in a rotary 'conditioner.' This is dried in a rotary drier and passed to a rotary cooler, then screened to remove the desired size fraction of granules. The undersize and crushed oversize are returned to the cycle. The drier exhaust gases are scrubbed in a wash tower.

This plant has a nominal capacity of 12½ tons/hr., and was supplied and erected by Edgar Allen & Co. Ltd.

Bagging and loading plant

The two units for packing triple superphosphate or granular compounds into open-mouth sacks are rated to have a combined capacity of 1,000 1 cwt. bags/hr. They are built at first-floor level to enable road and rail transport to be loaded by gravity chutes. Any oversize material is first screened out and disintegrated, after which pre-weighed batches are automatically fed to the filling spouts. Passing along a band conveyor the bags are closed by Union Special sewing machines, and then proceed either to the loading chutes or to the bagged material store. Provision is also made at the same location for bulk products from the triple and single superphosphate and compound stores to be loaded to railway trucks.

Storage buildings

There is a variety of different storage buildings constructed for the reception of the raw materials and the finished products.

As has already been mentioned, the materials are in every case brought into the storage buildings by conveyor belt. However, the method of extraction from the stores varies for process reasons from building to building, and is in some instances by mechanical shovel; in others by overhead travelling grabbing crane, and so into hoppers served by conveyor belt; and in others again by drag scraper equipment to a sub-floor hopper served by conveyor belt and elevator.

The relevant costs of the stores were, of course, dependent upon the quantity stored—the larger the quantity the smaller the unit cost—but also they were much influenced by the method of extraction.



Store for superphosphates in bulk.

The least expensive method was by drag scraper, as this arrangement facilitated the utilisation of an 'A' frame building which conformed closely with the shape of the heap of stored material, so avoiding waste space internally and reducing wind loads externally. Such a building is the phosphate rock store, with a capacity of 20,000 tons.

The superphosphate fertiliser and the compound fertiliser stores, with capacities of 22,000 and 20,000 tons, respectively, are 58½ ft. from floor to eaves, and each of them is a twin-bay building. The bays are 60 ft. in width, separated by a 20-ft. wide central conveyor bay, and are in one case 280 ft. long and in the other case 320 ft. long. The material is abstracted from each of these stores by two 5-ton overhead travelling grabbing cranes which discharge into six elevated bunkers.

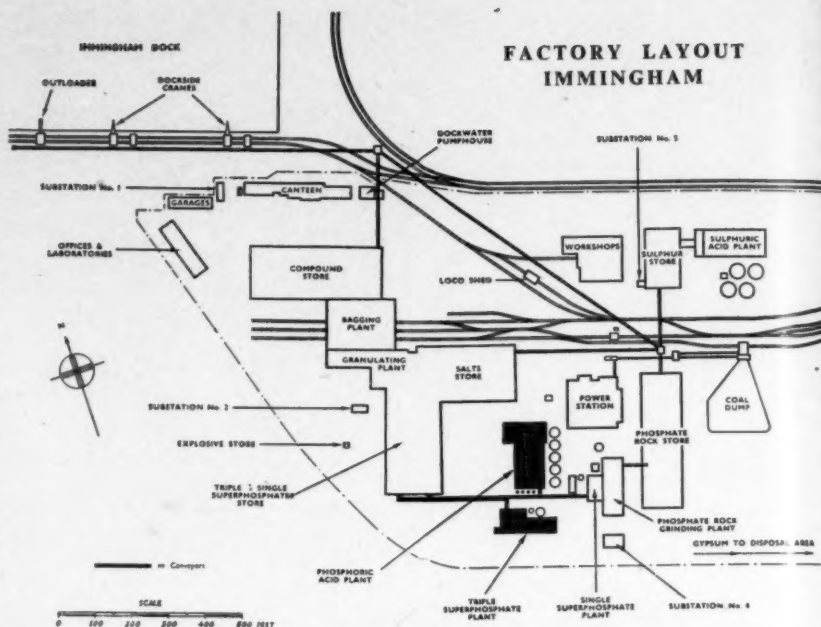
The salts store, with a capacity of 10,000 tons, is a three-bay building, each of the side bays being 60-ft. span and the centre bay 30-ft. span. The store is divided into storage bins by 15-ft.-high reinforced concrete walls, extending along three sides of each bin. The fourth side adjacent to the centre bay of the building is left open to allow access for the mechanical shovels, by means of which the material is abstracted from the bins and discharged into the batching plant hoppers at the end of the building.

The sulphur store of 8,000 tons capacity is similar as regards method of operation to the salts store, but has only one single bay of 100-ft. span, within which the sulphur is stored and the mechanical shovels operate.

Utility buildings

The most important of the utility buildings is, of course, the power house. This building comprises a boiler house, 114 × 72 ft., accommodating four super Lancashire boilers now, and with space for a fifth one to be installed in the future; and a generator room, 105 × 75 ft., accommodating three W. H. Allen steam turbines, each driving a Bruce Peebles 1,750-kW alternator. There is space within this room reserved for the installation in the future of a fourth turbo alternator set, the foundations for which have already been constructed. Also in this building is a water treatment section, 72 × 23 ft., accommodating a 3,500-gal./hr. Permutit water treatment plant, a switchroom, 58 × 22 ft., and a number of small rooms occupied by chlorination plant and emergency batteries, offices, etc.

Each of the four super Lancashire boilers has a steaming capacity of 12,500 lb./hr. at 260 lb./sq.in. and 700°F. Each boiler is fed from a 40-ton overhead coal bunker through a chute fitted with a Romer Lea recorder. The boiler is fitted with a Hodgkinson coking stoker and Cope's feed water regulator. The ash is extracted from the grate by hand and scraped into a submerged ash conveyor, which takes the ash



The factory is integrated by a 2½-mile conveyor system.

outside the building and into the elevated hopper, from which it can be removed either in rail wagons or road lorries. The instrumentation for the boilers was installed by Thermocontrol Installations Co. Ltd., and this deals not only with the boilers themselves, but also with the incoming steam from the waste heat boilers in the sulphuric acid plant which, under full working load, provide some 25,000 lb./hr. of steam. The boilers are served by forced-draught and induced-draught fans and the hot gas is passed into an elevated reinforced flue and so to the brick chimney. This flue is 150 ft. long and is constructed in 18-ft. bays within the building and a 38-ft. bay outside.

The steam produced at 250 lb./sq.in. from the boilers and sulphuric acid plant is required primarily for process purposes, but before putting it into the process buildings it was considered economical to utilise it for the generation of the factory electrical supply, and it was, therefore, passed through the steam turbines, the full load ratings of each of which is 33,000 lb./hr. Each turbine can pass out 18,000 lb./hr. of steam at 15 lb./sq.in., and this pass-out steam is then used in the process and also for space heating throughout the factory.

The condensers are cooled with water from Immingham Dock, which is introduced into the building in a 21-in.-diam. pipe run from the dock water pumphouse to the power house.

In the turbo-generator room there are also accommodated, in addition to the generators themselves, two 7,500-gal./hr. feed water pumps, one of them steam operated and the other electrically operated, a 75,000-lb./hr. deaerator and three 2,000-gal./min. booster pumps, which boost dock

water to a 110-ft. head, for circulation to the several gas-washing towers on the site and also to the fire hydrants.

The water-treatment plant was supplied and erected by the Permutit Co. and comprises a Spiractor unit with brine tank and twin filters and chemical pumps and boiler feed conditioning apparatus.

The cooling water is supplied to the turbo-generator room from the dock water pumphouse situated some distance away, but the pumps are operated by remote control at the central control cubicle in the turbine house. In the dock water pumphouse there are three 4,500-gal. min. W. H. Allen vertical-spindle centrifugal pumps which are fed from the dock through twin Glenfield & Kennedy rotary screens and automatic float valves.

In this building are also to be found two small debris pumps which pump the debris collected from the screens back into the dock, and two Gwynne surface-water pumps, one of them good for 2,500 gal./min. and the other one good for 1,000 gal./min. These latter two pumps pump all the surface water collected on the site into the dock, but on occasions of excessive flow the collector sump is allowed to overflow into the dock-water sump and so the rain water enters the cooling water system. The cooling water main, which feeds not only the power house but also the sulphuric acid plant, is 33 in. diam. and as it leaves the dock water pumphouse, it is dosed with chlorine as a protection against mussel growth.

It was found necessary to install a pumping system for the surface water, in view of the fact that the high-water level at Immingham Dock and indeed in the Humber Estuary is only 2 ft. below the finished floor levels of the factory build-



The factory as it neared completion at the end of 1950.

ings and 18 in. below the factory road level. A gravity system was, therefore, not practicable.

Material handling

The conveyor system in the factory is $2\frac{1}{2}$ miles long and is made up of 68 different belts varying in width from 20 to 42 in. and running at speeds varying from 15 to 230 ft./min.

The main incoming conveyors are designed to handle 320 tons/hr. from the dockside to store, one of these conveyors being 850 ft. in length. Belt weighers are installed at key points in the system, and maximum light and access for maintenance has been achieved throughout.

Centralised control is provided for the outgoing conveyors in a specially ventilated control room with a mimic diagram and indicator lights showing the layout of conveyors diagrammatically. Any of a number of sequences can be selected and fully interlocked, and visible and audible warnings are relayed to the quayside and principal working points of the system. This automatic interlocking is very important, as it acts as a safeguard against a serious pile-up of material in the event of a failure occurring at any point in the particular sequence in use.

On one particular conveyor the gantry is provided with a hot-air drying and ventilating system installed by Brooks Air & Heat Systems Ltd., in order both to dry out the product and maintain good working conditions which otherwise would be impaired by the vapour given off by the material.

Each of the materials carried on the belts has different properties—abrasive, corrosive, hygroscopic or just sticky—which call for varying designs of the idlers and

a variety of different treatments at the delivery points, some of the materials being removed from the belts by normal type throw-off equipment and others being ploughed off by scrapers.

Coal, brought into the factory by rail trucks, is discharged into a hopper by a 20-ton Rotaside wagon tippler, supplied and installed by Strachan & Henshaw Ltd. From this hopper, coal can be fed by inclined conveyors to either the power house bunkers, a road bunker or to a swivelling chute discharging to a coal store. This has a capacity of 8,000 tons of coal, which is marshalled by drag scraper. The reclaiming is effected by reversing the drag scraper bucket and discharging the coal into a hopper which is alongside the wagon tippler hopper, both of which are served by the one conveyor system, capable of handling 75 tons/hr.

The elevated enclosed galleries in which the conveyors are housed, where they run between the quayside and the buildings presented a few problems in design.

On the quayside, where space was restricted, the outgoing conveyor had to be positioned over the incoming conveyor and the gallery housing. The former is, therefore, cantilevered from the supporting steel trestles so as not to interfere with the feed from the travelling intake hoppers through the roof of the lower gallery, and so as to facilitate discharge of the finished product from the upper gallery to the travelling outloader feeding the ships. The majority of the lattice girders in these galleries are 61 ft. in span and are usually 8 ft. in depth, except where travelling throw-offs are accommodated, when the depth is increased to 13 ft. One span is 154 ft. long and it has to support at its centre the ends of inclined lattice

girders from a sloping gallery. Another span is 161 ft. in length, one end of it being made deeper than the other so as to accommodate a belt weighing mechanism.

Care had to be taken to allow provision for expansion of the galleries due to temperature changes, as such expansion must be controlled so that no lateral displacement of the gallery occurs, as this would destroy the true alignment of the belt and so cause it to run off the idlers.

Principal contractors and sub-contractors

Granulating plant: Edgar Allen & Co., Ltd.
Granulating plant erection: B. & A. Engineering Co. Ltd.
Turbo-alternators and Pumps: W. H. Allen Sons & Co. Ltd.
Rubber lining: Andre Rubber Co. Ltd.
Quayside cranes: Sir William Arrol & Co. Ltd.
E.O.T. cranes: Babcock & Wilcox Ltd.
Broadfield plant: Bradley Pulveriser Co. Ltd.
H.T. cables: British Insulated Callender's Cables Ltd.
H.T. switchgear, transformers and motors: British Thomson-Houston Co. Ltd.
Structural steel: Cargo Fleet Iron Co. Ltd.
Power and lighting: H. J. Cash & Co. Ltd.
Stone facings: Constons Ltd.
Chemical plant: Dorr-Oliver & Co. Ltd.
Rubber lining: Dunlop Rubber Co. Ltd.
L.T. switchgear: George Ellison & Co. Ltd.
Conveyor gantries: Alex Findlay & Co. Ltd.
Diesel locomotives: John Fowler & Co. (Leeds) Ltd.
Piling: Franki Compressed Pile Co. Ltd.
Evaporators: W. J. Fraser & Co. Ltd.
Acid-resisting brickwork: General Refractories Ltd.
Water mains and L.P. steam plumbing: Matthew Hall & Co. Ltd.
Heating: Hinton Jones (Engineers) Ltd.
Grinding and screening plant: International Combustion Ltd.
Glazing: W. G. Kaleyards Ltd.
Lagging: William Kenyon & Sons Ltd.
Tankwork: Mechans Ltd.
Asbestos sheeting: Medway Roofing Co. Ltd.
Main building contractors: A. Monk & Co. Ltd.
Plant for triple superphosphate: Ernest Newell & Co. Ltd.
Shuttering: Pochins Manchester Ltd.
Sulphuric acid plant: Simon-Carves Ltd.
Boreholes: F. Smith & Son (Grimsby) Ltd.
Conveyors and lagging plant: Spencer (Melksham) Ltd.
Concrete water and effluent pipes: Stanton Ironworks Co. Ltd.
Hoppers and doors: Staveley's Ltd.
Power station pipework: Stewarts & Lloyds Ltd.
Landskrona filters: R. O. Stokes Ltd.
Chemical plant erection: Sturdy Engineering Ltd.
Lead flashing, gutters and downpipes: Henry Tattersall & Co. Ltd.
Power station instruments: Thermocontrol Installations Co. Ltd.
Painting: Turner & Brown Ltd.
Railway track: Thos. W. Ward Ltd.
Motor control gear: Watford Electric & Manufacturing Co. Ltd.
Power wiring: F. H. Wheeler (Sheffield) Ltd.
Windows and doors: John Williams & Sons (Cardiff) Ltd.

Tablet machines used to make ceramic products

Tablet machines are now being employed in the ceramics industry for producing insulator parts, insulator beads and sparking plug insulators. The clay powders are compressed by the dry method, resulting in very large outputs of products with accurate dimensions. Some form of treatment is required with the powders, it being necessary to incorporate a binding agent and to form the powder into granules.

According to Manesty Machines Ltd., manufacturers of tablet machines, the following method could be used: Process by wet granulation method, using sodium silicate mixed with a little water as the moistening agent. For 1 lb. of material use 4 fl. oz. of sodium silicate in 2 fl. oz. of water. Add a little more water if necessary to moisten thoroughly. To dried granules (size as required) use $\frac{1}{2}$ to 1% magnesium stearate.

For insulating beads a good powder to use is magnesium oxide. Process as above.

DISTILLATION

Theory, calculations, packed columns, azeotropic, extractive, batch processes

By H. H. Jones, B.Sc.Tech., A.M.I.Chem.E.

ADVANCES during the past year in the unit process of fractional distillation were not of a fundamental nature. The most valuable service rendered to the art in this period has been the publication of several textbooks summarising the mass of material, both practical and theoretical, that has been published in the past ten years. The subject has become so wide during this period that critical collation was overdue.

First among these books must be listed the fourth and entirely revised edition by Gilliland¹ of the well-known work which was formerly written with the late Prof. Robinson as the co-author. Gilliland has completely rewritten it and added many new chapters. There is, for instance, a greatly extended treatment of tray design and azeotropic and extractive distillation are treated very clearly. Also, the number of worked examples has been increased.

The second edition of Kirschbaum² in German has appeared, the treatment being similar to that in the former work, though many chapters are enlarged; in particular the thorough treatment of packed columns is kept well up to date. There is also a considerable appendix on vapour liquid equilibrium for many binary and some ternary systems, probably a better compilation than is to be found in any comparable work. Such data have also been published by Chu.³

The third edition of Perry's 'Chemical Engineers' Handbook'⁴ was another notable book of the past year. The enlarged section on fractional distillation shows that a largely successful effort has been made to condense and evaluate much of the information published during the past decade. The theory underlying most of the recent advances is incorporated in an admirably succinct manner. As in Gilliland's work, the new handbook places greater emphasis on the solution of some difficult design problems and these are so presented that the practising chemical engineer will have little difficulty in applying the methods.

An entirely new work, 'Unit Operations,'⁵ should receive attention if only for its exposition of bubble tray mechanics.

Laboratory- and pilot-scale operations are better served by two other comprehensive works that have been published

recently. The first of these, by Carney,⁶ 'Laboratory Fractional Distillation,' will be helpful to anyone who has to construct a small-scale column for an experimental investigation. A longer work, 'Distillation,'⁷ being volume 4 of the 'Technique of Organic Chemistry,' is also available. It is concerned primarily with batch distillation and is much wider in its scope than Carney's book. It develops in considerable detail the fundamental theory as at present understood and, in addition, the technique of laboratory work is given much space, with valuable suggestions for investigatory procedure of non-ideal systems such as the use of extractive distillation.

Theory

In a gratifying number of instances, contributions on the fundamentals of the subject were rather outside the previous well-trodden paths and, in particular, the amount of work published on batch distillation leaves one to hope that this particular operation will become better understood in the near future.

A new approach to the problem of sharpness of separation in binary batch distillation has been studied by Cichelli *et al.*,^{8, 9} who have introduced the idea of 'pole height' being equal to the product of the slope of the distillation curve at $x_D = 0.5$ and the fraction of the charge remaining in the still at this time. Using this concept, equations are developed relating the number of plates and the reflux ratio to the sharpness of separation in binary batch distillation when the usual simplifying assumptions including zero hold-up are made. It is possible by the application of this method to design batch distillation columns for any separation of binary mixture when the relative volatility is known.

Rose *et al.*¹⁰ present evidence that increase in hold-up does not always reduce the sharpness of separation in batch fractionation. It appears from this analysis that there is a critical reflux ratio below which greater hold-up will actually give improvement. The same workers¹¹ have also published a simple arithmetical step-by-step method for calculating a batch distillation curve. There is a definite similarity between the calculated curves

and those obtained by experiment. In both cases considerable hold-up is assumed. However, the procedure is rather more laborious than the normal plate-to-plate multi-component continuous method and this is realised by the authors, who suggest that it should be possible to simplify it. Nord¹² has developed graphical methods for the calculation of simple binary distillation for non-ideal solutions by a modification of the Rayleigh equation. The same method can be applied to flash vaporisation and an indication is given of the treatment of a multi-component mixture. Lloyd¹³ compares batch and continuous stills for a given separation and concludes, as might be expected, that a continuous still will make more efficient use of a certain number of plates than a batch still using the same column.

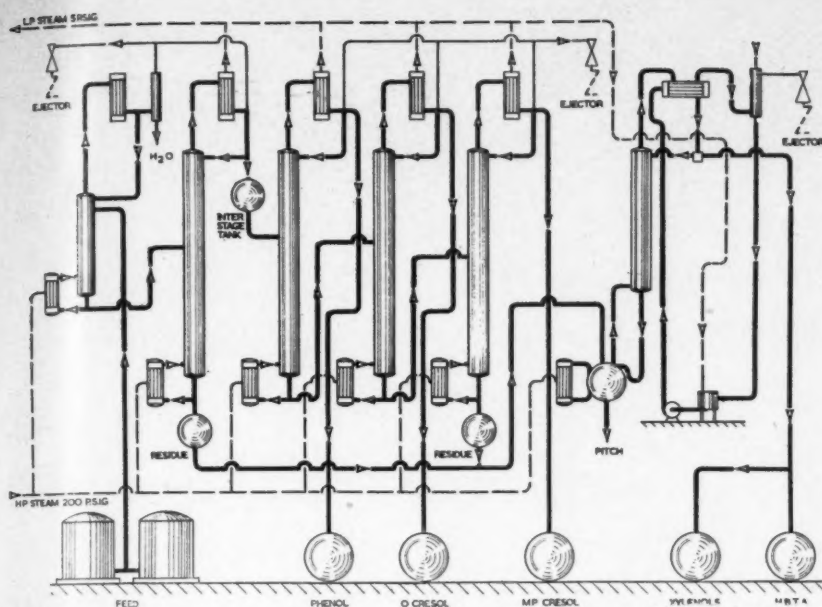
Calculations

In the realm of distillation calculation for multi-component mixtures, the equations of Underwood^{14, 15} have been rearranged by Alder and Hansen.¹⁶ The modification enables a fairly rapid solution to be achieved and, by successive approximation, this solution may be made more accurate. The usual simplifying assumptions are made and normally the distribution of the two key components is fixed, leaving that of the remaining components to be assumed. A series of calculations is made with different proportions between the rectifying and stripping plates. Murdoch¹⁷ has suggested a simpler mathematical attack, in which he defines 'doublet separations' as those with only two distributed components. Therefore the equations are relatively simple, but a trial and error method must be adopted. The use of punched card digital computers is suggested by Rose and Williams¹⁸ and, while such machines could be very useful for plate-to-plate calculations in cases where the usual simplifying assumptions may or may not apply, their cost would in most cases be prohibitive, except where they would be in constant use.

Weil¹⁹ goes further and describes the use of electronic computers at Northwestern University, U.S.A., to solve multi-component distillation problems where the usual simplifying assumptions do not hold.

Yu and Coull²⁰ give a graphical correlation to ascertain the number of theoretical plates necessary for a given separation when the reflux ratio and minimum plates have been determined. This correlation is somewhat similar to that of Gilliland,²¹ but as the plot is an arithmetic instead of a logarithmic one, the possibilities of error in reading off are reduced. Donnell and Cooper²² present another curve, again based on minimum reflux and number of plates. The relationship is practically a straight line, but some doubts have been expressed as to the clarity of the derivation.

Edminster²³ has extended the use of absorption and stripping factors to multi-component columns, while the calculation



Continuous tar acid distillation plant for the separation of pure products from coke-oven or gasworks crude material as installed in several British coal by-product works. The plant can be designed for various throughputs.

of the number of plates for multi-component mixtures is suggested by Coulson and Fyffe.²⁴ The method is somewhat similar to that of Scheibel and Montrose,²⁵ in that it postulates the prior calculation of a pseudo equilibrium curve based on the key components only. In this particular instance a calculation is made by plate-to-plate investigation at total reflux. Chu²⁶ uses Smoker's equation to evaluate a batch column where reflux ratio is continuously increased during rectification. Shira *et al.*²⁷ survey the previous methods for the calculations of minimum reflux ratio for multi-component mixtures and point out that they can be exact only by alteration of the method recently published by Underwood^{14, 15} and, in any case, it is only under conditions of two components being distributed between the top and bottom products that such will apply. A new method based on the Thiele and Geddes²⁸ plate-to-plate calculation is introduced and can be used for the distribution of greater than two components, variable molal overflow and variable relative volatility. The method, as may be imagined, is laborious and hardly suitable for plant design.

May,²⁹ on the other hand, has evaluated an equation for minimum reflux ratio which is precise, though time consuming, except for particular feed conditions. It does, however, go to the other extreme in postulating that in a head product there is no component present lighter than the light key and in the bottom product no component present heavier than the heavy key. This means that all components are distributed. From a practical aspect, however, it is probably more valuable because of this restriction than those based on two-component distribution only.

A nomograph for the solution of the Van Laar equations is given by Chu and Tung.³⁰ A method for the precise calculations involved in the separation of hydrocarbons is given in a series of papers by Hutchinson.³¹ No new basic principles are advanced, but the actual process-plant calculations that are necessary are set out in great detail and a simplified calculation form is suggested. This advice will obviously be of use in petroleum engineering, as it facilitates much of the repetition work. A rather ingenious use of the concept of relative volatility is employed by Fowler³² to calculate the number of plates for the production of high-purity products. In the solution of a binary problem the reverse volatility is employed which will give an operating line above the equilibrium curve. Logarithmic coordinates are employed and the step-wise procedure of McCabe and Thiele can be carried out on a considerably expanded scale, for that end of the curve under consideration.

Basic data

There was again a welcome increase in the amount of basic data on which distillation calculations must be based. The range of investigation has been widened to include conditions not previously considered but which sometimes arise in a large scale. It is hoped that efforts will continue to be made to relate laboratory determination to plant problems.

Othmer³³ describes an equilibrium still that has been developed for determination of vapour liquid equilibria at super-atmospheric pressure up to 125 lb./sq.in. Plots of activity coefficient and relative volatility against the logarithm of the molal

percentage show consistency of results. The work was undertaken to obtain basic data for a method for the azeotropic production of absolute alcohol previously described by Wentworth *et al.*³⁴ The still, however, could be used for the examination of other systems and, with the increasing use of high-pressure distillation, no doubt it will find wider application. Further work with this still is described by Moeller *et al.*³⁵ The data were obtained on the binary system ethyl ether/ethanol and the ternary ethyl ether/ethanol/water for the design of a column employing ether as an entrainer.³⁴ The actual information obtained on a plant scale confirms the reliability and usefulness of the still. An equilibrium flash still for use at high temperatures was described by Smith *et al.*³⁶ The design of the still was based on the need for accurate and reliable distillation data. Mercury vapour is used as the heating medium, and this ensures uniform temperature in the heated sections. With the present design of still any temperature between 400 to 800°F. can be obtained. The use of this still should open up possibilities of obtaining vapour liquid equilibrium data for high-boiling materials. The determination of vapour liquid equilibrium of partially miscible liquids is carried out in a still described by Smith and Bonner.³⁷ Vapour liquid equilibria at sub-atmospheric pressure for various systems are given by Bachman *et al.*³⁸ The systems were chosen as examples of those with markedly different relative volatilities and which also suggest the possibility of ideal behaviour. The variation of the relative volatility with pressure was calculated and the vapour pressure/temperature relationships for some of the components were also investigated.

The composition of vapours from boiling ternary solutions was investigated by Karr *et al.*³⁹ The system investigated was that of acetone, chloroform and methylisobutylketone. As acetone and chloroform form an azeotrope and it was considered that the third component would give a possible means of extractive distillation, the investigation was carried out to determine the ternary data. It was found that this ternary data could be predicted from the three binaries with only 0.5% mol. deviation in the vapour composition between the calculated curves and those obtained by experiment. The method of calculation was that of Redlich and Kister,⁴⁰ which in turn was based on the theoretical principles enunciated by Wohl.⁴¹

The system of methyl ethyl ketone and secondary butyl alcohol was investigated by Amich *et al.*⁴² The ability to separate this binary is useful in the manufacture of petroleum chemicals and the vapour liquid equilibrium was, therefore, of importance. It was considered that the data could be obtained in a still developed by Carlson and Colburn,⁴³ fitted with an adiabatic jacket. Data were obtained at two pressures and, if corrections are made for activity

coefficients, the data appeared to satisfy within experimental error the requirement:

$$\int_0^1 \log \frac{Y_1}{Y_2} dx = 0$$

Again, the same x/y data can be expressed with good agreement by the method of Clark.⁴⁴

The volumetric and phase behaviour in a methane/hydrogen sulphide system has been investigated by Reamer *et al.*⁴⁵ The estimation of equilibrium data from total pressure data by graphical integration of the Duhem equation for non-ideal systems is proposed by Orlicek.⁴⁶ Haldenwanger⁴⁷ gives two equations for determining the relative volatility depending on whether the mixture is of polar or non-polar substances.

Data on azeotropic systems are becoming more readily available as investigations proceed.

Lecat^{48, 49} gives details of over 400 azeotropic systems not previously collected. In addition, Horsley^{50, 51} has published lists. With the advances in the field of petrochemicals it is becoming increasingly important to separate hydrocarbons of different homologous series and, in this respect, details of azeotropes and non-azeotropes, between hydrocarbons and acetonitrile, are reported by Bishop and Denton.⁵² The range of hydrocarbons includes aromatics, olefines, paraffins and naphthenes. As a result of this work it was proposed that acetonitrile may be used as a separating agent. On theoretical grounds by Kuhn and Massini⁵³ it was deduced that an azeotrope will be formed when the difference in the boiling points of the pure components is less than the heat of mixing divided by 5.4.

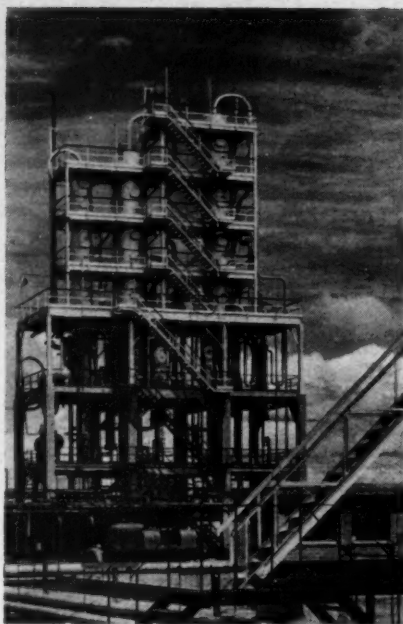
Equilibrium constant changes for light hydrocarbons, which are of great value in the separations necessary in petroleum refining, are presented by Smith and Smith.⁵⁴

Coming to more theoretical data, a method for determining the thermodynamic consistency of vapour/liquid equilibrium data is given by Steinhäuser and White⁵⁵ and by Gilmont *et al.*⁵⁶ who also gives a simple method of determining activity coefficients from relative volatility.

Packed columns

Probably the best collection of data on the operation of packed columns is still to be found in the work by Kirschbaum,² who has extended his previous investigations to include new types of packing made of metallic gauze. There has also been further information published by Koch and van Raay⁵⁷ on a modified Stedman packing which is less efficient than the original, but easier to assemble in a column.

In a series of papers published by Hands and Whitt,^{58, 59, 60} various aspects of packed column design and operation are considered. In the first of these, the design of a column is based on a rather different concept of performance from the



The distillation unit of the Vacuum Oil Co. refinery at Altona, near Melbourne, Australia. This refinery was built at a cost of £A1,000,000 and commenced production in July 1949.

flood point accepted previously. The operating rate is that at which the vapour velocity is just high enough to cause entrainment. It is assumed that when this stage is reached the vapour tends to rub the liquid off the packing and hence mass transfer is impaired. The formulae derived as the result of experiments so based and with different packings and test mixtures have been used for calculating entrainment rates for a number of columns. Comparison with operating rates which were found to give satisfactory performance on these columns indicates that the approach was justified. In the second of these papers, film resistance during rectification was investigated, and it was found that the liquid film resistance is controlling, which generally supports what little other work has been published on this subject, and is the opposite to what has already been found with wetted wall columns. The third paper deals with the calculation of the height of a suitable column by reverting to the use of the H.E.T.P. concept, because of the limitations imposed by the newer H.T.U. method. A formula is derived for the H.E.T.P. which is dependent only on the reflux rate, diameter of the packing and the viscosity of the liquid. This is a result of correlations of published information, and it will be noted that vapour velocity and wall effect do not appear in the formula. For this reason it is simple and differs markedly from others which have been published, such as that of Kirschbaum.⁶¹

The effect of vacuum operation on the efficiency of packed columns shows that there is little variation over the range of

normal pressures. This work is reported by Berg and Popovac⁶² and by Struck and Kinney,⁶³ but the latter paper has been criticised on the ground that the experiments at low pressure were carried out with a different test mixture from those at atmospheric pressure.

The hydrodynamics of packed columns have been investigated in more detail by Reed and Fenske,⁶⁴ with particular reference to the phase densities at the point of flooding.

A series of papers has been published by Pratt relating to the design of packed columns for batch distillation and absorption. The first⁶⁵ of these gives special attention to optimum column diameter and determination of packed height, while such practical problems as packing supports and liquid distribution are investigated. In the second,⁶⁶ separation of binary and multi-component systems is considered, together with the calculation of batch distillation curves. In the third,⁶⁷ values of H.T.U. and H.E.T.P. have been calculated for two systems, in order to illustrate the influence of variations in throughput on the performance of columns packed with various sizes of rings and saddles. The construction and arrangement of packed columns with particular reference to the practical points of packing supports, reflux distribution, etc., and their limitations as compared with bubble cap columns are given.

A suggested set-up for an automatic packed column distillation plant is outlined.

In the last paper⁶⁸ an example illustrative of the design methods proposed in the other three is given. The binary mixture chosen is the non-ideal one of methyl acetate and water.

Azeotropic and extractive distillation

Colburn⁶⁹ has comprehensively reviewed the design basis for azeotropic and extractive distillations, with particular reference to the theoretical consideration on the presence of a third component on the activity coefficients. He shows that azeotropic and extractive distillation calculations may be conveniently made by the use of some of the procedures found useful for multi-component distillation generally, but account must be taken of the effects of the high liquid load and the deliberate non-ideality of the systems. Recent data show that the liquid film resistance is greater than is normally the case in straightforward distillation and hence the efficiency of the plates is lower. There are results which show that this efficiency is normally of the order of 25 to 30%.

Details have been published on the efficiency of columns separating C_4 hydrocarbons by extractive distillation with furfural by Grohse *et al.*⁷⁰ and by Engs *et al.*⁷¹; methylamine is employed as the selective solvent.

A separation of xylenes from mixtures with non-aromatics by the use of pyridine

as an entrainer is reported by Foxon⁷² and in another paper⁷³ he instances the use of 2-dimethylaminoethanol; in both cases an outline of plant performance is given.

If, however, an extractive instead of azeotropic method is used with nitrobenzene as third component, Woerner⁷⁴ states that it is necessary to distil 10 to 20% of the circulating solvent to remove gum formed during distillation to avoid accumulation in the system.

A rather different approach to the problem of batch azeotropic distillation is made by Berg *et al.*,⁷⁵ who investigated the effect of charge composition on the recovery of constituents during an azeotropic distillation in a packed column. Five different systems were investigated and it was found that the percentage recovery of bottoms product is greater than with the separation carried out without the entrainer.

The quantity of the more volatile component recovered at a certain purity will be increased, but improvement in this purity is not always obtained.

An interesting application of azeotropic distillation is the dehydration of methyl ethyl ketone⁷⁶ where, by the utilisation of increased pressure, the azeotropic composition is so altered that the product on condensation separates into two layers. The M.E.K.-rich phase is refluxed to the pressure distillation zone, M.E.K.-free of water being withdrawn as bottom product. The water-rich phase is distilled in a second column to yield the normal M.E.K./water azeotrope, which is returned to the first column.

An industrially important application of azeotropism, is claimed by the Standard Oil Development Co.,⁷⁷ where a separation of ethanol from propanol and higher alcohols is achieved by the addition of a relatively large amount of water as reflux to the stripping zone. By this means dilute ethanol substantially free from impurities is withdrawn as bottoms.

The use of azeotropic distillation combined with continuous esterification for the removal of water is described in another patent.⁷⁸

Batch distillation

A good introduction to beginners in this subject is a review of the fundamental principles and techniques by Given.⁷⁹ This is quite exhaustive and collects together in a good descriptive manner the various theories and experimental work on which they are based. The most important recent advances in this field have been summarised elsewhere in the present review.

Large-scale distillation equipment

Attempts have been made to place the investigations of efficiency of industrial plates on a more rational basis. Williams *et al.*⁸⁰ studied the efficiency of plate columns in the separation of eight binary systems. The plate efficiencies obtained in these experiments are given. The results

can be correlated with plate efficiency as a function of the product of viscosity and relative volatility for reflux ratios up to 1.5. However, at higher reflux ratios this agreement no longer holds. The quantity of reflux flowing down the column also has an effect, but may be expressed as some function of L/V .

The correlation of bubble plate efficiency with viscosity is further extended by Grunberg and Nissan⁸¹ to bring in critical constants. The viscosity can be determined from these with an accuracy of about 10%, and a nomograph is provided for this purpose.

Of particular interest in plant-scale fractionation is a paper by Uitti⁸² suggesting that, to obtain good separation, top temperatures are not a sufficient measure of the purity of the overhead product; he also makes some suggestions as to the location of the control point somewhere lower down the tower. Two articles by Davies^{83, 84} investigate in some detail the design of bubble cap trays involving consideration of various factors influencing plate efficiency, such as tower diameter, tray spacing, cap design, etc. These are fixed before the actual calculation of the tray layout, and equations and recommended values for liquid gradient, pressure drop, slot velocity, etc., are given. These two papers will repay further study by anyone faced with the problems discussed in them.

An investigation into fractionating tray performance of a bubble cap tray 16½ ft. in diameter is described by Rhys.⁸⁵ Various different bubble cap arrangements and holding-down methods are investigated and, as a result, valuable recommendations are made on the design and method of liquid distribution over large bubble cap trays. Equations are deduced from the results for vapour velocity. The performance of commercial fractionators is described by Gelus *et al.*⁸⁶ Here the investigations were carried out on columns under actual working conditions, because the units could not be taken off stream. The various steps to be followed in obtaining the necessary information are set out. An example is given of a column preparing, as overhead, dimethylcyclopentane from petroleum naphtha. Calculations performed by the method of Lewis and Matheson⁸⁷ showed that an overall efficiency of 35% was obtained, as is to be expected in an extractive distillation.

An important condition for the operation of industrial equipment is the pressure drop across the bubble tray, this frequently being the limiting factor in the operation of vacuum columns. Chu⁸⁸ compares methods that have been proposed for the calculation of this figure. He describes those by Rogers and Thiele,⁸⁹ Kirkbride,⁹⁰ Dauphine⁹¹ and Eld⁹² and concludes that Dauphine's is probably the soundest. In this a correlation has been made of extensive data from numerous experiments and it also breaks down the pressure drops into

three components. Chu limits his investigation to columns operating at a normal range of vapour velocity.

Further information has been published on the tower packing in the form of corrugated trays of multi-layer expanded metal lath.⁹³ This paper by Schofield reported performance characteristics at various pressures compared with conventional bubble cap towers. It is stated that at atmospheric pressure an existing tower containing 36 bubble cap trays at 1½-ft. spacing, on being replaced with this packing, gave an equivalent of over 100 bubble trays while working at twice the original loading.

Laboratory fractionations

Williamson⁹⁴ has made a useful survey of up-to-date laboratory fractionating practice and deals with various types of columns, head design, still heating methods, etc. The layout, testing and operation of Dixon gauze columns and spinning band columns are described.

The more restricted field of rotating columns is covered in another review,⁹⁵ which deals with the general theory as well as reviewing the experience of the U.S. National Bureau of Standards with such units.

A particularly exhaustive treatment of the methods to be employed in the laboratory fractionation of tar acid mixtures is given by Jäger and Kattwinkel.⁹⁶ The columns used and the techniques applied are described in detail and the results presented with admirable clarity.

Information has appeared on the design of new types of fractionating stills for laboratory work. Benner *et al.*⁹⁷ describe a rotary condensing fractionation vacuum still which can operate under lower absolute pressures than bubble caps or packed towers. It is stated that with binary test mixtures 18 theoretical plates are obtained in a 32-in. tower. The latter consists of a central spinning cooled surface rotating in a vacuum space, and pressure drops of 0.2 and 0.5 mm. are reported. To avoid leakage under the conditions of operation, a special motor drive integral with the column is employed. A rotary thermal rectifying column is reported by Bryan *et al.*,⁹⁸ which is an elaborate device consisting of an outer heated jacket and an inner condenser tube which is rotated. Vapour moves across the annular space and condenses on the inner surface, from which it is thrown out by centrifugal force to form part of the reflux stream. It is emphasised that enrichment takes place primarily from vaporisation and condensation effects and not from mass transfer. The main advantage is that it can be worked at a lower absolute pressure than more normal columns. It is stated that a column 1 m. long and 45 mm. outside diameter contains about 15 theoretical plates when calculated by the Smoker method. It would appear, however, that there are certain

mechanical difficulties in the operation of such a unit. The paper develops the mathematical analysis of the functioning of the column based on a previous paper by Bowman and Briant.⁹⁹

Laboratory fractionating columns are sometimes run by the system of intermittent reflux, i.e. a long period of total reflux, followed by a short interval during which there may be low reflux or total product only. The efficiency of such a method, as opposed to the more normal one of continuous working, is investigated theoretically by O'Leary *et al.*¹⁰⁰ Certain assumptions are made and it is shown that the main distillate composition is always heavier than that obtained by continuous reflux at the same mean reflux ratio. The difference is small, however, under normal conditions. Experimental work is quoted which confirms this deduction.

A high-efficiency laboratory fractionator is described by Dixon¹⁰¹ where the packed column is surmounted by a specially designed condenser to facilitate the condensation and removal of high-melting-point materials.

A fully automatic laboratory unit is described by French¹⁰² in which the column containing 100 theoretical plates separated 89% thiophene from crude coke oven benzene. It is claimed that the distillation period can be cut considerably by the use of this unit.

A similar claim is made for a rotating glass column as described by Irlin and Bruns.¹⁰³ This considerably cuts the time needed for laboratory distillations by eliminating the period for approaching equilibrium. Kincannon and Manning¹⁰⁴ show that in a laboratory column the number of components present did not affect the efficiency of the column as calculated from a binary mixture.

The operation of the Podbielniak Hyd- Robot column is described by Starr *et al.*¹⁰⁵

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Handling of carbon black. (B.S.

1714:1951.) A committee of the British Standards Institution is trying to specify methods to be adopted to reduce the difficulties in handling carbon black and B.S.I. has now published Part I of B.S. 1714, 'Recommendations for handling carbon black—Loading and stowing in ships.' As soon as possible further recommendations will be published for the unloading and inland transportation of carbon black and it is also hoped to publish minimum quality standards for the packaging of this substance. (Price 1s.)

Copies of this standard may be obtained post free at the price stated from the British Standards Institution, 24 Victoria Street, London, S.W.1.

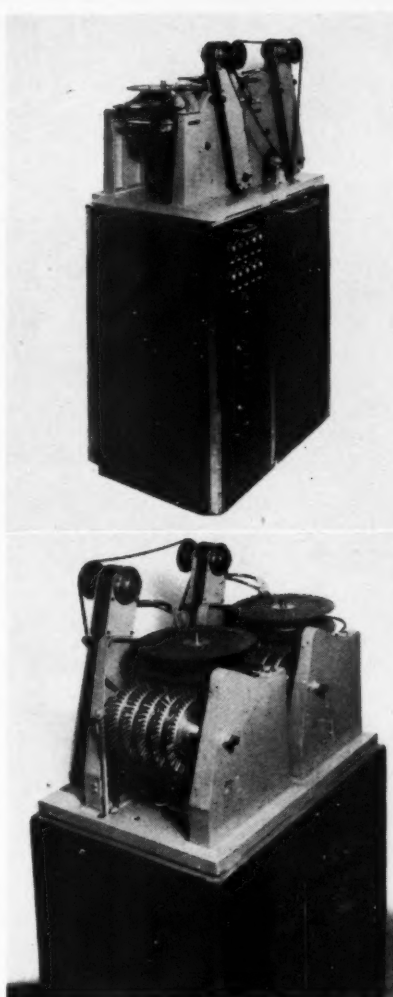
A Mechanical Analogue for Separation Problems

Within the last two years a number of papers have been published in the United States on the use of computing machines for the solution of distillation calculations. With one exception these reports concerned the use of standard computing machines, punched card or electronic, the exception being an electrical network device which was specially designed to carry out plate-to-plate calculations. In a paper presented at Leeds to the Institution of Chemical Engineers by Dr. J. S. Forsyth, N. L. Franklin and H. Winning, Jnr., of Leeds University, a mechanical analogue of a stagewise distillation process was described and a pilot model representing a two-plate separation on a five-component mixture was demonstrated. Here is a summary of this paper.

THE calculating machine differed from those previously described in that the separation process is replaced by an equivalent mechanical process rather than by a series of mathematical equations. The flow streams of vapour and liquid which are interchanged between plates in a normal column are represented in quantity and composition by an equivalent interchange of steel balls between the mechanical stages. The balls employed differ in size, so that the total flow can be represented by the number of balls and the composition by the proportion of balls of the various sizes. On a given plate the plate liquid is represented by such a stream and a mechanical unit, which is provided with information on the relative volatilities of the components involved, is employed to derive from this stream a smaller stream representing the vapour in equilibrium with the plate liquid. From the vapour stream so formed a portion is withdrawn and fed to the plate above. The residual vapour stream, together with that part of the plate liquid stream which was not used in deriving the vapour, is combined with incoming liquid and vapour streams from adjacent plates. Under equilibrium conditions this combined stream is identical in composition, except for statistical fluctuations, with the plate liquid originally considered. From this stream a portion is separated and fed as reflux to the plate below, whilst the remainder is recycled as plate liquid.

Details of construction

In a device of this type flow channels must be provided for the various streams and suitable elevators are needed to supply potential energy, but the important units are those in which the vapour stream is derived, and those in which samples of the vapour and plate liquid streams are withdrawn for dispatch to adjacent plates. The balls employed in the pilot model are of five sizes, differing by steps of approximately .001 in., the mean size being about $\frac{1}{16}$ in. diameter. The relative sizes of the balls are selected only on the basis of convenience in handling and separation, the properties of the components which are represented by the balls are incorporated in the mechanical units and not in the size of ball employed, so that by altering the



[Courtesy: Institution of Chemical Engineers]

Top: Overall view of the front of the calculating machine with its dust cover removed.
Bottom: A closer view showing the different parts referred to in the text.

programme of the machine the same balls can be used to represent any number of chemical components up to five.

Vapour stream unit

The unit which derived the vapour stream operates in two stages, the first of which consists of a ball sizer where the

plate liquid stream is separated into five substreams each consisting of balls of a single size. Each of the substreams is fed to one of five similar proportioning units in each of which a definite fraction of the total number of incident balls is deflected into a vapour stream while the remainder fall into a separate channel. The units differ in the proportions of the incident substreams which are deflected into the vapour channel. Since in a real separation process the vapour in equilibrium with a given plate liquid is given by

$$\varphi_A = \frac{\alpha_A x_A}{\sum \alpha x}; \quad \varphi_B = \frac{\alpha_B x_B}{\sum \alpha x} \dots \text{etc.},$$

$$\text{where } \sum \alpha x = \alpha_A x_A + \alpha_B x_B + \dots \text{etc.},$$

where the relative volatilities of the components are given by $\alpha_A, \alpha_B, \dots$ etc., the calculated vapour composition is unaffected if the latter are multiplied by a common factor. In the analogue this factor is so chosen that the greatest relative volatility is less than or equal to unity. The relative volatilities of the remaining components will be between zero and unity, and the term $\alpha_A x_A$ can be represented by taking from the plate liquid substream A a proportion α_A . If these proportions of the substreams are then combined the stream which is derived has exactly the composition of the vapour in equilibrium with the original plate liquid stream. The vapour stream so formed will in general be greater in quantity than that which should flow to an adjacent plate, and a device which involves a sampling process, is employed to meter off the correct quantity of this stream. By this means the correct quantity of balls having, in the statistical sense, the correct vapour composition is dispatched to the next plate, whilst the residual vapour stream is combined with that part of the plate liquid stream which was not used in deriving the vapour stream. Together these streams represent plate liquid minus vapour, so that if the reflux and vapour streams from adjacent plates are also combined with this stream it now represents the original plate liquid stream together with the material which must be dispatched as reflux to the plate below. It is assumed in the definition of a theoretical plate that this reflux has the

same composition as the plate liquid, so that a metering device is incorporated in this stream, which is divided into a reflux stream for dispatch to the plate below, and a recycle stream of plate liquid. Under equilibrium conditions the recycle stream is identical with the original plate liquid stream in quantity, and, except for statistical fluctuations, in quality. Since a complete column is built up from a number of similar plates plus feed, condenser, and still auxiliaries, a corresponding number of mechanical units can be combined to provide an analogue of such a column.

Operation

In operation the analogue plates are interconnected to represent the column or assembly of columns to be considered, side streams and multiple feed positions being introduced as required. The plates are then programmed according to the relative volatilities of the components to be separated, and to the reflux ratio which it is proposed to employ in the column or columns. The proposed feed stream or streams can then be introduced into the unit, and the product streams be analysed by counters.

Liquid composition

The composition of the liquid on any plate may be determined by counting the number of balls in each of the homogeneous substreams obtained from the plate liquid before the vapour stream is derived. In the present design of unit this information is required in order that adjustment to the reflux ratio and relative volatilities can be made on a plate for those problems in which these factors cannot be assumed constant throughout the column. In problems where the number of actual plates required for a separation is to be determined a knowledge of the plate efficiency is essential. If this information is available, and if the efficiency is the same for all components on a given plate, modification of the metering devices enables this factor to be incorporated in the analogue.

Possibilities of the machine

The two plate pilot model of the analogue, which has been constructed has been used to gain experience, and for cost and performance comparisons with alternative electrical analogues designed to solve similar problems. Whatever the ultimate form of the unit such an analogue, in addition to its use for practical calculations, would be a useful tool for theoretical studies. Since it does not require a preliminary estimate of the product composition in the form of operating equations before a computation can be carried out, problems such as multicomponent batch distillation calculations, with and without plate hold-up, should be soluble. This would permit a study of a subject which at present is intractable.

Effect of Added Components on Distillation

WHEN the volatilities of the components of liquid mixtures are approximately the same or when a constant boiling mixture is formed, distillation is generally carried out in the presence of added components. Investigations into the enrichment of vapour obtained by the addition of a third component were made for three binary mixtures: (1) water-ethanol, (2) water-nitric acid, and (3) water-formic acid, and the results are described by S. P. Samaddar and S. K. Nandi, Chemical Engineering Laboratories, Indian Institute of Science, in *Transactions of the Indian Institute of Chemical Engineers*, 1950, 2, pp. 29-35.

The added components were calcium chloride, glycerol, common salt and sucrose for the first system; sulphuric acid for the second; and silica gel, phthalic anhydride and nitrobenzene for the third system.

An Othmer type still was used for determining the enrichment of vapour, caused by the introduction of a third component in the feed mixture.

Water-ethanol system

The vapour composition became richer in alcohol by the addition of all the extra components. The enrichment obtained was mostly due to the depression of the vapour pressure of water caused by dissolved substances. In this work the maximum concentration of CaCl_2 used was 33.3% (200 g. feed mixture+100 g. CaCl_2) and this caused a boiling point rise of 8°C. Similarly with 33.3% glycerol, the rise was 3°C., with saturated NaCl 5°C. and with 50% sucrose 2°C.

Water-nitric acid system

Extremely high vapour enrichment was obtained by the addition of H_2SO_4 . The elevation of the boiling point for 30% H_2SO_4 by weight in liquid was 8°C., for 40% 16°C., 50% 26°C., 60% 41°C. and 70% 64°C. Water-nitric acid solution forms a constant boiling mixture, with maximum boiling point at 121.9°C. containing 68.5% by wt. HNO_3 , which means that on distilling acids of lower strength than the azeotropic mixture, the distillation is more dilute. Results showed that the azeotropic point shifted very widely from its normal position by the addition of H_2SO_4 .

Water-formic acid system

Silica gel does not appreciably alter the volatility of this system when the feed is dilute, but when the feed is concentrated the vapour becomes weaker in formic acid by the addition of silica gel. Phthalic anhydride enriched the vapour to a small extent. Nitro-benzene is immiscible with water thereby forming a pseudo-azeotropic

mixture and distribution of formic acid was more favourable to the water than to the nitro-benzene phase. Very little improvement was found when the third component was immiscible.

Packed column distillation

Rectification in the presence of the added components was also studied by the authors for the water-ethanol system as considerable vapour enrichment was obtained by the addition of third components to this binary mixture. For this purpose a small all-glass distillation unit was set up which consisted of a 3-litre still and a column $\frac{3}{4}$ in. diam. by 33.5 in. packed height and was operated under total reflux. The distillate composition became stronger in alcohol by the use of CaCl_2 or glycerol. Better results were always obtained when the third component was sprayed from the top along with the reflux than with the third component in the still.

Attempts to carry out rectification for the water-nitric acid system with sulphuric acid sprayed from the top proved unsuccessful because when the concentrated H_2SO_4 was sprayed from above the heat of reaction was very great, endangering the glass tube.

Laboratory distillation column

An American firm recently announced a new miniature *Hyper-Cal* laboratory distilling apparatus designed for the precise fractional distillation analysis of small volume samples of gasoline, light oils, fatty acids and other organic and inorganic chemicals.

All the instrumentation and controls necessary for distilling samples from 2 to 200 c.c. in volume, boiling over the temperature range of from -40° to 300°C. and from atmospheric pressure to as low as 2 mm. mercury absolute, are built into a panel assembly. Accuracy and ease of operation are said to be ensured by an automatic control of reflux ratio, of distillation take-off rate and of distillation pressure. A potentiometer of special range has been incorporated in the unit for accurate determination of distillate temperatures.

'I.C.E.' August

Articles include:

Centrifuging by E. Broadwell.

Vacuum Refrigeration by D. Wittenberg.

Radioactive Isotopes in Metallurgy.

Production of Essential Oils by Distillation

By Yves-René Naves, D.Sc.

(Givaudan et Cie., Geneva)

The author argues that the problem of essential oil production cannot be approached solely from the standpoint of distillation theories. His reasons for this view and suggestions for improving yields and arriving at a truly scientific basis for essential oil production are given in the following article.

IN most books and technical journals the production of essential oils by distillation in a current of steam is discussed exclusively with reference to thermodynamic laws. This approach is fallacious. The authors apparently regard such problems as involving solely the distillation in steam of a mixture of substances more or less soluble in water and more or less volatile. According to this reasoning, the problem is one of rectification of an essential oil, not of its production from vegetable material. In fact, the isolation of an essential oil from a plant by distillation in steam is influenced to a greater or lesser extent by two factors of prime importance:

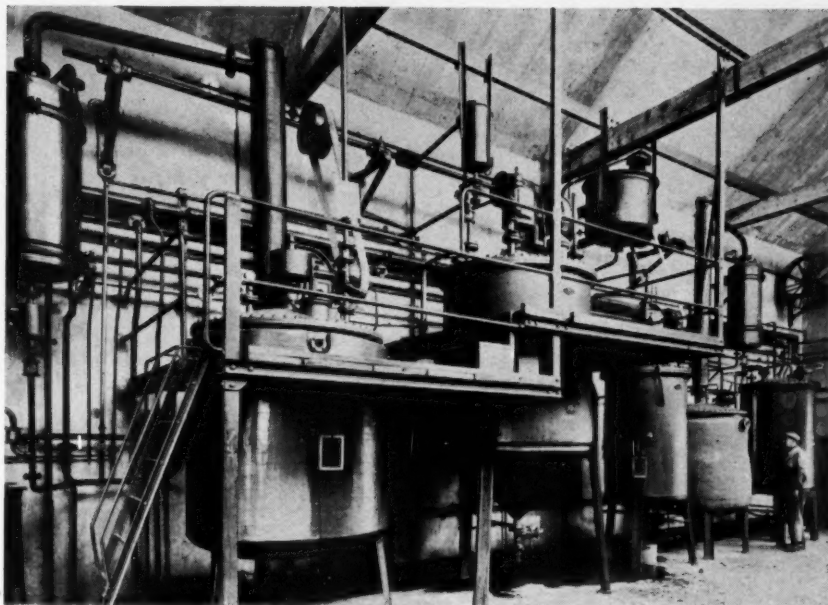
(1) Certain constituents of an essential oil are not readily amenable to distillation in steam, and they also vary among themselves in their degree of resistance to steam distillation, independent of their volatility in dry heat. Such constituents can only be distilled after modifications have been effected in the structure of the vegetable material during the operation.

(2) A certain number of the constituents are the products of chemical, mostly hydrolytic, reactions which likewise proceed during the operation.

It follows that the composition of successive fractions will deviate to a notable extent from that to be expected by applying the laws of thermodynamics and by an empirical study of the redistillation of the same essential oil in the same still and using the same procedure. Distillation of an essential oil from vegetable material differs from the operation of rectification in taking a longer period and requiring more steam, even when improvements are made in the design of the still and in the conditions of charging and packing the starting material. This point is clearly demonstrated by introducing into a still, at the start of distillation, a quantity of essential oil equal, for example, to that anticipated as the result of normal operation. The added oil is found to distil off very quickly, while the normal batch material requires much longer treatment. See, for instance, the behaviour of orris oil as described in *Helvetica Chimica Acta* (1949, 32, p. 1061).

We shall first consider the simpler case, in which all the constituents of the essential oil already exist at the start of distillation. The conditions of (1) above are then applicable, and the complicating effects of (2) are absent in most cases.

In these cases, and in the absence of noticeable proportions of constituents that



Steam-jacketed stills equipped with stirrers for distillation of volatile products at Givaudan's factory in Geneva.

are relatively very soluble in water, the composition of the essential oil obtained by distillation from the plant is identical, or very nearly identical, with that of the distillate of the benzene or petroleum ether extract from the plant. On fractionating the two distillates during operations, we find that corresponding fractions differ appreciably in composition.

Faulty conclusions may be drawn in such an eventuality if the distillation or the extraction is incomplete. This possibility should be borne in mind.

Usually the distillation of an essential oil from a plant is accompanied by chemical changes involving the conversion of non-volatile components of the vegetable material into 'essential' products and the destruction of volatile constituents. By contrast, the chemical changes are much less profound when the benzene or petroleum ether extracts are distilled, especially when such distillates are obtained by use of superheated steam under reduced pressure. The following examples demonstrate the difference between the composition of a distilled essential oil and that of the corresponding oil obtained by distillation (using superheated steam and reduced pressure) of the benzene or petroleum ether extract of the same plant.

Ylang-ylang oil

It was shown in 1930-32 by the present author and Glichitch (*Parfums de France*, 1930, 8, p. 350; 1932, 10, p. 41; see also Naves and Mazuyer, 'Natural Perfume Materials,' New York, 1947, p. 269) that the sesquiterpenes in which ylang-ylang oil is so rich are almost entirely absent from the distillates of the petroleum ether extract. They must thus have been formed, during distillation, from constituents of the flowers which are insoluble in petroleum ether. The relative proportions of these constituents and of benzyl benzoate in successive fractions obtained in course of production of the essential oil differ from those predicted from the laws of thermodynamics when these are applied to the case of an artificial mixture of sesquiterpenes and benzyl benzoate. The relative proportions likewise differ from the distribution taking place when the whole ylang-ylang oil is fractionated by rectification in steam. An eminent specialist in distillation problems once proposed that the whole distillate of ylang-ylang oil should be collected on the spot and later fractionated in Europe. The resultant fractions would probably be quite different from those traditionally expected by the perfume trade and obtained by fractiona-

tion during distillation from the vegetable material. This illustrates the point we made at the outset: the problem of essential oil production cannot be approached exclusively from the standpoint of distillation theories.

The observations of Glichitch and myself were confirmed by Traubaud (*Perfumery and Essential Oil Record*, 1937, 28, p. 406) and by Garnier and Defaud (Guenther, *American Perfumer*, November 1938, p. 44).

Clove oil

A very similar case was encountered by the author in the course of examination of the benzene extracts of clove buds (*Helv. Chim. Acta*, 1948, 31, p. 379), when the distillate from the extract was found to be practically free from sesquiterpenes, whereas the latter are present in high proportion in the essential oil obtained directly from the bud. Numerous other examples of the same phenomenon could no doubt be found.

Chemical reactions during distillation

The importance of the chemical transformations increases with increasing period of distillation, so that the effects of such changes can be reduced by using plant of small capacity and by processing smaller batches. These measures have been found to suppress the hydrolytic changes to a considerable extent, especially those involving esters and lactones. The improvement was shown to be particularly noticeable in experiments on the production of essential oils from lavender, clary sage, geranium, amber seed, celery seed, lovage root, etc. Essential oils produced in the small sizes of apparatus are relatively richer in esters and lactones.

Among the best known of the chemical reactions taking place during the isolation of essential oils by subjection of the plant to distillation are the cleavage of α -unsaturated aldehydes and ketones (the lidenic cleavage of Chelintzev). Hydrolysis of citral yields methyl heptenone, and that of farnesal yields dihydropseudoionone. These reactions are promoted by alkaline reagents such as lime water. In 1931 the author showed that these two aldehydes and the corresponding ketones co-exist in lemongrass oils. In 1907 Barrowcliff found pulegone and methyl cyclohexanone-3 in American pennyroyal oil (from *Hedeoma pulegioides*), and in 1942 Naves found the same components in European pennyroyal (*Mentha pulegium*). The oil from the latter also contains, as shown by the author in 1942, piperitenone and methyl-1-cyclohexen-1-one-3. Pennyroyal oil, however, also contains (Naves, 1943) methyl-1-cyclohexanol-3 side by side with the corresponding ketone, and it is impossible formally to demonstrate that this pair of ketones co-exists as a consequence of the lidenic cleavage. At least a portion of the lower ketone may have originated by biosynthesis.

In 1934 Pfau and Plattner found in

cedar essences, side by side with α - and γ -atlantones, acetyl-9-dipentene, acetyl-9-terpinolene and methyl-1-acetyl-4-cyclohexene-1. The three latter compounds may be regarded as products of the lidenic degradation. At about the same time, in collaboration with Rupe and Clar, they detected the following compounds in curcuma oil: tumerone and dihydrocurcumone, ar-tumerone and curcumone (or acetyl-9-*p*-cymene). More recently (Naves, 1948), methyl-1-acetyl-4-cyclohexene-1 (or tetrahydro- Δ^3 -*p*-methylacetophenone) and *p*-methyl-1-acetophenone were found in the essential oils of cabreuva (*Myrcarpus fastigiatus* and *M. frondosus*) together with terpenic and sesquiterpenic constituents. Here the origin may be attributed to biochemical oxidation and dehydrogenation. This example illustrates the need for caution in attempting to trace the cause of lidenic cleavage.

Degradation of isothiocyanates to nitriles has been observed in the course of distillation. Thus the benzyl isothiocyanate in the essential oil of garden cress becomes degraded to benzyl cyanide. If the plant is not minced prior to distillation, the degradation product alone is isolated. Similarly, the phenylethyl isothiocyanate in the essential oil of watercress or the oil of American cress (*Barbarea praecox*) yields phenylethyl cyanide (Gadamer, 1899). Similar reactions may account for the evolution of hydrocyanic acid, hydrogen sulphide, carbon bisulphide and ammonia during the distillation of numerous essential oils, particularly those of umbelliferous fruits.

Other reactions involve the formation of non-distillable products, and may be initiated or continued in the 'hot' portion of the condensers and in the coating of 'resins,' and may result in the contamination of the essential oil.

The case of wormseed oil has been studied by a number of workers. Conditions for obtaining an oil of satisfactory quality, as established by Schimmel & Co., are short duration of distillation, use of small stills and a steeply sloping condenser, and running the distillate hot to obtain rapid separation of the oil from the water. In 1911 Nelson stated the conditions for efficient distillation as: use of superheated steam, avoidance of large-diameter stills, rapid distillation, hot running of the condensates and rejection of the distillation waters.

It has been noted that the dehydrocostus lactone of costus root, which constitutes a high proportion of the distillates from costus extracts (resinoids), is almost entirely absent from the essential oil. It probably undergoes polymerisation rather than hydrolysis.

The azulene of achillea (milfoil) essential oil is formed in course of distillation. Katherine Graham showed in 1933 that its mother substance is absent from the petroleum ether extract, whereas it can be extracted with chloroform.

Many of the condensations and polymerisations taking place during distillation are due to the reactions of philodienes.

Various distillation practices, such as cohobation, lead to the accumulation in the reaction zone of constituents that are soluble or readily emulsifiable in water, thereby increasing the destructive effect. Operating conditions and procedures that favour the refluxing of distillates should therefore be examined with a critical eye; likewise, procedures involving partial recycling of the distilled products. From this aspect the processes of distillation by entrainment with gases in a closed cycle, so tempting on thermodynamic grounds, may prove unsatisfactory with respect to the composition and value of the distillates.

Improving yields

The key to improved yields of distillates may reside above all in making the essential constituents more accessible to the action of water and steam. Independently of modifications in the design, structure and dimensions of apparatus, this objective may be realised by suitable mechanical treatment of the vegetable material, for example, comminution, crushing and ultrasonic disintegration. But such treatments may initiate reactions before or during the distillation which prove detrimental to the composition and quality of the essential oil, so that each case calls for a close analytical study.

An alternative approach to the problem involves the addition to the charge of surface-active materials capable of modifying the processes of liberation of the essential products and of promoting the homogenisation of the phase in which the water vapour is generated or into which the water vapour is injected. Such procedures do not appear to have been adopted so far on a large scale. Here again the possibility of concomitant chemical changes must not be forgotten.

Conclusions

The isolation of essential oils from vegetable materials will not be established on a truly scientific basis until we know the exact location in the plants of the constituents and the changes to which they are susceptible after contact with boiling water, as well as the nature of the other plant constituents and of the products of their transformations during distillation. The other facet of the problem, i.e. the phenomenon of distillation itself (or, more precisely, the application of theories of the steam-distillation of heterogeneous mixtures of sparingly water-soluble substances and the associated modification of distilling equipment), has recently been studied with considerable success. Bearing in mind, however, the complexity of the requirements and the absence of precise data, it will be necessary to resort to a rigorous empirical method based upon an exhaustive analysis of the yields and of the nature of the constituents.

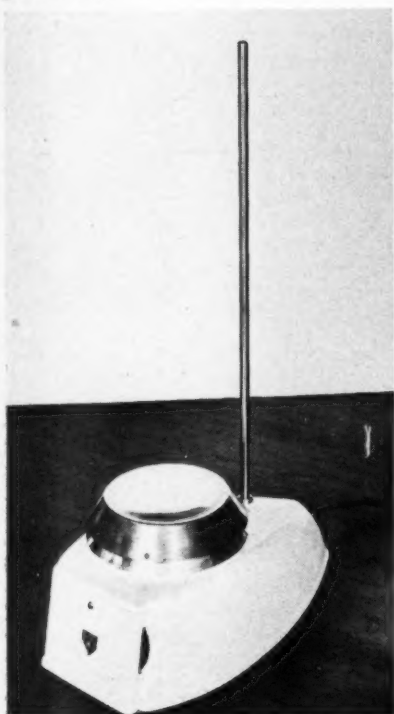
New Trends in Instruments and Instrumentation

Many of the most ingenious devices of the scientific instruments industry have been created to meet the many special problems of academic and industrial chemistry. Thus, the first British Instrument-Industries Exhibition which opened at Olympia, London, for eleven days from July 4, has much to hold the attention of the chemist and the chemical engineer. This impressive display shows the enormous progress made in improving conventional instruments and in devising new ones. Particularly notable are the many devices for automatic control and the advances made in process plant instrumentation. Among the newer techniques, much has been achieved in applying nucleonics to industrial instrumentation. Several innovations in laboratory apparatus and equipment are also to be seen. Here is a summary of some of the outstanding exhibits from the viewpoint of the chemist and chemical engineer.

Baird & Tatlock (London) Ltd. displayed the largest number of items they have ever shown at an exhibition under the four main headings: (1) general chemistry; (2) pathology, bacteriology and allied sciences; (3) industrial equipment; and (4) special apparatus.

A recent development is a **magnetic stirrer hotplate** consisting of a cast base of light metal alloy to which is fitted a stainless steel retort rod and the hotplate. The stirrer itself consists of a small length of magnetic material completely sealed in glass so that the metal does not come into contact with the solution which is being agitated.

Other exhibits in the chemical section included **balances, centrifuges, water distillation apparatus, laboratory ovens, potentiometric titration apparatus** and a range of **volumetric and sintered glassware**.



[Photo: Baird & Tatlock Ltd.]

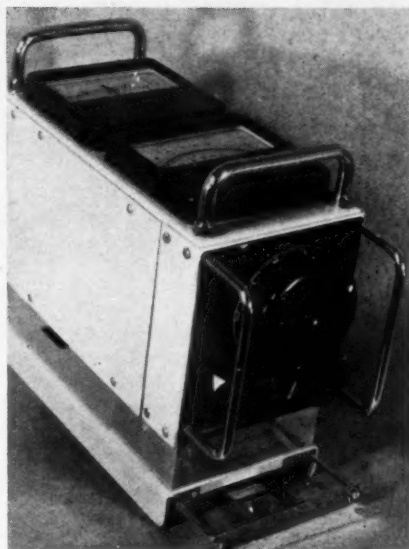
Magnetic stirrer combined with hotplate.

Among many exhibits in the industrial section were **coal testing and gas analysis apparatus**, a **Redwood centrifuge**, a wide range of standard apparatus such as **coal ashing furnaces, vapour pressure apparatus, viscometers, a metallurgical polisher and electrolytic deposition equipment**.

Baldwin Instruments Co. Ltd. showed, among other items, a **beta ray thickness or weight gauge** for measurement of the thickness or weight of paper, board, plastics, metal foil, etc. A radioactive isotope with a useful life of four years is used with an ionisation chamber operating the indicating mechanism calibrated in units of thickness or weight. The A.C.-operated model is being used extensively in industry and there is a battery-operated portable laboratory model. A **gamma ray thickness or weight gauge** marketed by the same company is for use with materials of greater thickness and weight than can be measured with beta rays. A gamma emitting isotope is used.

Also shown was the *Baldwin N.P.L. moisture meter*, which operates on the principal that the dielectric properties of most organic substances, and of some inorganic ones, vary with the amount of moisture they contain. A low-frequency current is passed through the dielectric being tested and the circuit is arranged to respond partly to the resistance and partly to the capacitance of the sample, in such a proportion as to give the best scale of moisture content. Calibration charts for most cereals, seeds and dehydrated foods are available.

Bristol's Instrument Co. Ltd. This firm's exhibits included systems for the indication, recording, control and telemetering of such variables as temperature, pressure, flow, liquid level and humidity. In the **pressure spring thermometer**, of which the Series 500 is the latest development, the primary element translates the temperature changes it experiences into pressure changes, which are transmitted through capillary tubing to a pressure spring measuring element which moves a pen arm across a chart. The Series 500 **recording thermometer** is available with liquid-filled, vapour pressure or gas-filled measuring systems. The pen arm



[Photo: Baldwin Instruments Co. Ltd.]

Portable β -ray thickness gauge for laboratory and inspection department use.

swings across a rotary chart which is driven at a convenient speed by a synchronous motor or clockwork. As many as four independent recording mechanisms, all tracing on the same chart, may be installed in one case when it is convenient to have a direct comparison of several variables. The Series 500 **controllers** are air-operated, using the free vane principle of automatic control, and require an air supply at 20 lb./sq.in. For more complex control problems, 'reset integral' control, in which an integrating action is added to the proportional action to compensate for load changes, or 'derivative' characteristics where a correction proportional to the rate of change of the controlled variable, or a combination of these may be followed.

Cambridge Instrument Co. Ltd. exhibited a comprehensive range of **temperature indicators, recorders and controllers** for temperatures from -200°C . up to the highest met in industry or research. There was also a series of **pressure gauges** having dials 4, 6 and 8 in. diam. and covering ranges from -15 to $+10,000$ lb./sq.in., as well as a variety of pressure recorders. **Gas analysis apparatus** shown included

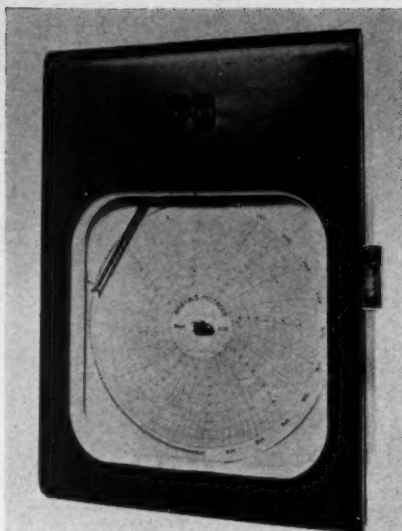
instruments for the measurement of CO₂ and CO in boiler flue gases, dissolved oxygen recorders, furnace atmosphere controllers and exhaust gas testers. A re-designed **direct-reading pH meter** was on show. Recorders and automatic controllers of pH were also available. Another new instrument which has wide possibilities in industry is the **electronic quick-acting recorder**, which has a response speed of only 0.5 sec. and can be employed for the accurate reading of any rapidly changing phenomena, which can be translated into terms of electrical voltage or resistance. Other exhibits included **electro-titration apparatus**, **vibrographs**, **surface and interfacial tension apparatus**, etc.

Chance Bros. Ltd. exhibited scientific and optical glassware, including *Hysil* standard chemical-resistant flasks, beakers, test tubes, etc. An all-glass syringe, which is available with interchangeable parts, was also shown. Another interesting item was the **Flamemaster hand torch** for light welding and glass working. This torch weighs only 12 oz. and can be used with gas/air or gas/oxygen. Several interchangeable flame units are available and it has a built-in leak-proof economiser. Agents are *W. Edwards & Co. (London) Ltd.*

E. K. Cole showed several 'atomic' devices. One was a **radioactive thickness gauge** which operates by measuring the absorption of radiation from a radioactive source. As no contact is made with the sample, a wide variety of materials can be measured, including soft materials and wet or sticky coatings. Another device on display was a **radioactive ore detector** weighing only 3½ lb. (1.5 kg.). It has no controls, no electronic circuitry and no interconnecting cables except for headphones and can be carried by hand.

A. C. Cossor Ltd. exhibited a variety of instruments for use in industrial and research laboratories. The Model 1049 **double-beam industrial oscillograph** was developed to measure phenomena of zero or very low frequency and to make photographic records of transients requiring a high photographic writing speed. The oscillograph incorporates the Cossor 4-in. flat-screen double-beam tube type operating at 2 kV, but provision is also made for operation at 4 kV when maximum spot brightness with hair-line focus is required. There was also an **oscillograph camera** designed for use with oscillographs, including Model 1049. It provides a simple means of recording oscillograph pictures, either by taking single shots of a stationary waveform or it can be used for continuous records of non-recurrent waveforms and slow transients by the moving film method.

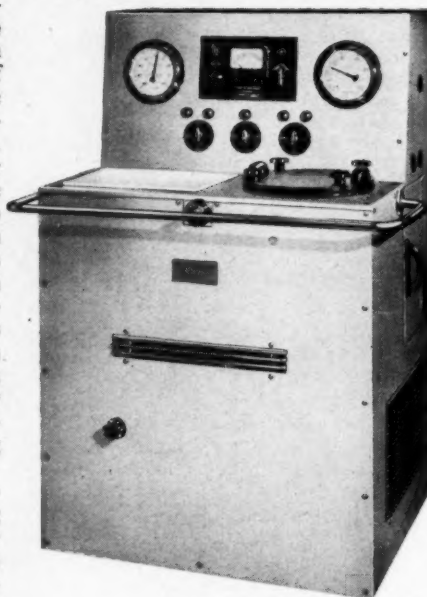
Cinema-Television Ltd showed many interesting electronic instruments, including an industrial **electronic metal detector** which automatically detects metal, ferrous or non-ferrous, in any non-metallic material. A second model exhibited was a portable metal detector with oscillator for



[Photo: Bristol's Instrument Co. Ltd.]
Recording thermometer available with liquid-filled, vapour-pressure or gas-filled measuring systems.

the accurate location of buried metal or pipes, etc., which, it was claimed, will trace the routes of pipes buried to a depth of 30 ft. Among 'atomic' instruments on this stand was a personal protective radiation or monitor **dosimeter** for detecting gamma radiation and hard x-rays.

Coley Thermometers Ltd. exhibited dial distance-reading **temperature indicators** of vapour pressure and mercury-in-steel actuations, in sizes from 2½ to 8 in. diam. There were also examples of these instruments fitted with electric contacts designed to give visible or audible signals at pre-set temperatures. A demonstration panel showed an indicating temperature controller and a temperature recorder con-



[Photo: W. Edwards & Co. Ltd.]
Freeze drier.

troller, maintaining the temperature of water within fine limits and recording the latter on a chart. The **gas temperature controller**, which is available with a mercury-in-steel indicator or with a vapour pressure indicator, will control the temperature of gas-heated apparatus to within fine limits. The instrument comprises a 4-in. dial indicator fitted with electrical contact mechanism which controls a small solenoid valve, operated by the mains supply and contained in the case with the indicator. Mercury-in-steel indicators can be supplied in ranges from -40° to +1200°F. or equivalent Centigrade. Other exhibits were **vapour pressure indicators** claimed to be suitable for ranges from -40° to +450°F.

D.S.I.R. instruments. Some 30 instruments developed by the research associations and establishments of the Department of Scientific and Industrial Research were demonstrated. They included mechanical, optical, hydraulic and electronic instruments. Certain of the items offer possibilities of a wider usefulness than that provided by their original purpose and are available for marketing by any interested manufacturer.

Of direct interest to the chemical industry were an **automatic constant-volume fraction collector** and a **pressure-controlled thermostat** developed at the Chemical Research Laboratory, Teddington. The former collects a constant volume of any liquid, independent of its characteristics. Rising in the collecting vessels, the liquid interrupts a beam of light and so operates a turntable when the required fraction has been obtained. The thermostat operates on the principle that the control of the pressure over a boiling liquid gives a fine control of temperature. It was developed in connection with studies of the corrosion of steel.

A **gas-content tester** in which small samples of molten metal cool under reduced pressure, so increasing the size of the gas bubbles given off and thus giving a more accurate result than otherwise obtainable, was shown by the British Non-Ferrous Metals Research Association.

A **liquid flowmeter** designed to measure predetermined weights of liquids and to dispense them at constant head was shown by the British Internal Combustion Engine Research Association.

A **dew-point meter** for the investigation of corrosion by small quantities of sulphuric acid in the flue gases of water tube boilers was exhibited by the British Coal Utilisation Research Association.

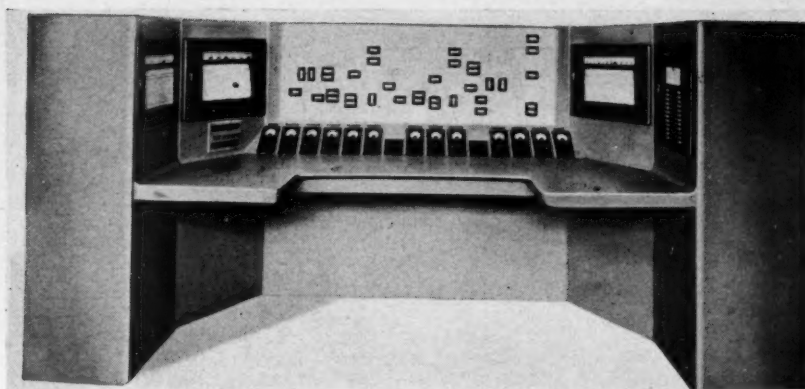
W. Edwards & Co. (London) Ltd. showed their range of **Speedivac rotary pumps**. The single-stage and two-stage models attain pressures as low as 0.0005 mm. Hg and 0.00005 mm. Hg respectively. For lower pressures a Speedivac diffusion pump is employed, the various models giving a vacuum of better than 5×10^{-6} mm. Hg and a special self-fractionating model, reaching better than 5×10^{-7}



[Photo: Griffin & Tatlock Ltd.]
Microd 'Atomill' for laboratory use.

mm. Hg. One of the latest additions to the Speedivac rotary pump range, a two-stage high efficiency unit for research and educational laboratory work, is a small compact pump which, because of its exceptional vacuum performance, can be employed for industrial applications where high speeds are not required and space is limited. Its ultimate vacuum is 0.0001 mm. of mercury and the displacement 0.8 cu. ft./min. Supplied together with a $\frac{1}{4}$ h.p. electric motor the pump occupies a space of only $15\frac{1}{2} \times 9\frac{1}{2} \times 11\frac{1}{2}$ in. Another Edwards' exhibit was the Model 3 **centrifugal freeze dryer** specially designed to produce blood-plasma in a preserved and readily transportable form. It can be also used for drying many other biological liquids. Drying is accomplished without raising the temperature to a height that would lead to denaturation of the material by freezing solid in a vacuum and drying thereafter by sublimation. Originally an objection to this technique was the considerable ebullition of the plasma to the point of freezing, leading to a great increase of bulk. This has now been overcome by the technique of centrifugation, until the plasma becomes a solid, which completely suppresses any tendency to ebullition.

Elcontrol Ltd. specialise in electronic 'on-off' or two-position controllers, designed particularly for industrial as distinct from laboratory use. Their field of application is wide since industry is calling for increased automatic control of repetitive operations and processes. The exhibits included the following standard items: **Liquid level control equipment** which gives automatic control of pumps and valves for emptying and filling liquid containers, tanks, boilers, etc., and which can be used with a very wide variety of liquids and foams. It is actuated by the level of the liquid itself. The two versions of **furnace safeguard equipment** give immediate fuel cut off on flame failure in gas and oil fired furnaces respectively. The units are not only safety devices, since they greatly reduce explosion risk, but they also lend themselves to incorporation in fully automatic ignition controls giving push-button start, and can readily be linked with



[Photo: Evershed & Vignoles Ltd.]
The Centroller, a self-contained unit for centralised operation of a process plant.

temperature controllers where required. The Elcontrol range of **process timers** included one calibrated in seconds (alternative ranges to 10 or 100 sec.), uncalibrated delay switches and a continuous cyclic timer having independently variable on and off periods. Repetition resistance welding depends on accurate timing and a non-synchronous **electronic weld timer** was shown fitted to a standard welding machine. Electronic on-off **temperature controllers** actuated either photo-electrically or by a thermocouple or by a resistance thermometer unit have been developed, and examples of complete equipments were shown.

Electronic Instruments Ltd. showed a wide range of **pH meters and electrodes** including the Model 30 battery portable, Model 23 direct-reading pH meter and the Model 28 which is specifically designed for automatic industrial pH control. One arrangement enabled the visitor to vary at will the pH of a continuous stream of water and to observe the result on an outside indicator. In the field of precise **resistance measurement** by simple direct-reading instruments were shown the Model 47 milliohmmer which ranges from a fraction of a milliohm upwards, and at the other end of the scale a Model 29 megohmmeter which extends into the 20,000,000 megohm region. Two new 'atomic' instruments were a **beta-ray thickness gauge** which depends upon the absorption of beta particles by the material under test, and a **vibrating-reed electrometer** which is a commercial development of a design produced at A.E.R.E. for the measurement of small ionisation currents. Finally, this stand displayed a **smoke detector** which depends for its action upon the absorption of alpha radiation by smoke particles. It is claimed to be instantaneous in action and contains no moving parts or thermionic tubes.

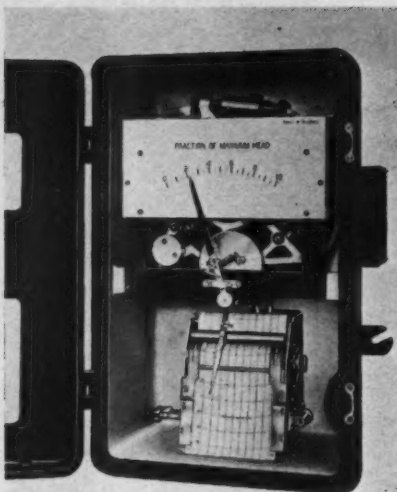
Electrothermal Engineering Ltd. displayed laboratory heating equipment including **heating mantles** for 50- and 100-litre flasks and beakers, conical flasks and Buchner funnels. A new development, which resulted from research into the difficulties associated with the heating of

fractionating columns, is the flexible and elastic **heating tapes** as described and illustrated in INTERNATIONAL CHEMICAL ENGINEERING, June, 1951, p. 287.

Evershed & Vignoles Ltd. The principal exhibit was a self-contained unit for **centralised operation of a process plant**. Known as the 'Centroller,' it comprises a console or desk in which are concentrated the many controllers and recorders necessary for efficient operation of such plant. In addition, the Centroller has a control diagram having a number of miniature edgewise indicators inserted at appropriate points of measurement. Some of these have double pointers, one indicating the actual measured value, while the other shows the desired value set by the control setting equipment. Centralised control is achieved by an electronic system, which facilitates remote indication and control of flows, levels, pressures and other measurements. All measurements are transmitted to the Centroller in the form of small D.C. currents acting as electrical signals, thus permitting the use of multi-pen recorders and repeat indicators. The various process controller components are mounted on trays accommodated in racks behind the console face and can be examined from the back of the Centroller. Rapid servicing is facilitated, as the trays are coupled by multi-pin plugs, and may be withdrawn individually for inspection or replacement, all similar units being interchangeable.

The actual Centroller exhibited was designed for the centralised operation of a chemical process plant and contains 13 controllers, 5 for flow, 6 for liquid levels and 2 for pressure. Three six-point multi-pen recorders, 1 multi-point temperature indicator and 25 miniature edgewise indicators show the level and pressure in various columns and vessels, the flow in various pipes and the temperature in reboiler units.

Foster Instrument Co. Ltd. The range of **temperature measurement equipment** available covers the entire field of temperatures likely to be encountered in industrial or research work. Among the exhibits were single- and multi-point temperature indicators and recorders for use



[Photo: George Kent Ltd.]
Specific gravity recorder.

with both thermocouple and resistance thermometer elements, automatic temperature controllers, optical and radiation pyrometers for temperatures up to 3,000°C., a portable potentiometer and pyrometer tester and a pyrometer testing furnace. A feature of the **electronic indicating temperature controller** instrument is that the control mechanism operates instantaneously upon any departure of the temperature from the pre-set value, as opposed to the intermittent operation which characterises the mechanical type of controller. **Foundry pyrometers** for aluminium, brass, bronzes, etc., up to 1,300°C. and for high-content nickel alloys and ferrous metals up to 1,650°C. were demonstrated, in conjunction with the Teloscale large-scale illuminated indicator. The Teloscale, which was primarily designed for foundry use, can be read with accuracy from a considerable distance, so that a furnace operator can read his temperature without having to leave the casting bed.

Fielden (Electronics) Ltd. showed a comprehensive range of industrial indicators, recorders, controllers and telemetering equipment. The **Titragraph** is a completely new instrument which automatically graphs titrations. The chart of the recorder is turned according to the volume of liquid delivered by the burette, and the pen is deflected by the current derived from a pH meter. By using the **Tektor level control** it is possible to control the level of liquids or granular solids without any actual contact with the substance being controlled. The instrument has also been used as an interface detector, froth detector, counter, laboratory temperature control, etc. The **Manograph** was shown as an **air flow recorder**. It is available with full-scale deflection corresponding to as low as 0.25 in. water gauge, thus making the loss in head negligible for normal applications. Another version, the **steam flow recorder**, was also shown; this

model can be used at static pressures up to 250 lb./sq.in.

Foxboro-Yoxall Ltd. exhibited a selection from their range of **industrial control instruments**, comprising indicators, recorders, controllers and transmitters for temperature, humidity, pressure, vacuum, liquid level, flow, specific gravity, etc.

The principal exhibit was the new Consotrol series of **miniature control instruments**. This equipment has been developed and especially designed for use in the graphic or line diagram representation of process operation, but may also be mounted in the more conventional type of instrument panel with considerable saving in space. The new equipment was exhibited under operating conditions controlling a demonstration process and mounted in graphic panel form. A flow controller maintained constant the feed to the column and a level controller maintained the pre-set conditions within that column. A pressure indicator showed clearly the working pressure. The load on this process could be varied by changing the set point of the feed flow controller and, following this change, the level controller held the same level with a greater or lesser pass-out flow.

A. Gallenkamp & Co. Ltd. showed a wide range of laboratory apparatus. The **electrical apparatus** included the latest patterns of drying ovens, centrifuges, incubators, tube and muffle furnaces, controlled humidity conditioning ovens, hot-plates, stirrers, water baths, a photo-electric colorimeter, micro-melting point apparatus and Kjeldahl nitrogen apparatus. Other exhibits included Technico volumetric glassware, sintered filtration apparatus and standard joint glassware. Several types of **thermostatic water baths** were available, one being a single-walled glass vessel for a nominal maximum temperature of 100°F., but capable of successful operation at 140°F. Another was double-walled, comprising one glass vessel inside another, and designed for a maximum temperature of 210°F. Both can be fitted with a cooling coil for circulating a cooling liquid when the ambient temperature rises above the required setting. The accuracy of control was said to be within $\pm 0.025^\circ\text{F}$. at all temperatures and approaches $\pm 0.01^\circ\text{F}$. at 100°F. Both models can accommodate five viscometers, but they can be used for other applications requiring accurate temperature control. **Electro-titration apparatus** on display is supplied as two separate units, consisting of a titration stand and a mains-operated potentiometer. Stirring is almost noiseless and there is a choice of two fixed speeds from a stepped pulley on the motor shaft.

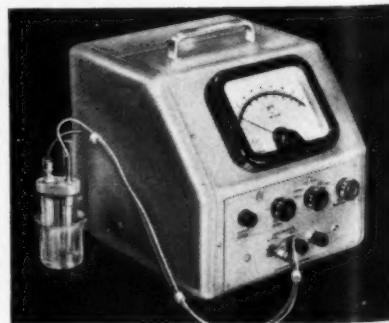
General Electric Co. Ltd. showed a comprehensive display of **switchboard and portable indicating instruments** in the form of voltmeters, ammeters, wattmeters, power factor meters, frequency meters and synchroscopes, in all grades and sizes. There was a demonstration of the mag-

netic sorting bridge, with special presentation on a large cathode ray tube; also a demonstration of the G.E.C. **photo-electric photometer and colorimeter**. Instruments for the measurement of mechanical quantities were represented by the **reed vibrometer** and the laboratory and pocket **layer thickness meters**. Light measurements were covered by a range of G.E.C. illumination meters and exposure meters. Remote indication and control instruments were also displayed.

Griffin & Tatlock Ltd. showed instruments and apparatus covering a wide range of operations in the chemical laboratory, including new and redesigned models. Among them was the **Microid Atomill**, which can deal with a wide variety of substances, pulverising by impact and attrition friable, crystalline, fibrous, resinous and other materials to products as fine as 300 B.S. mesh. Other exhibits covered operations as varied as stirring, shaking, drying, sterilising, gas and fuel analysis, metallurgical analysis, weighing, etc.

Honeywell-Brown Ltd. This firm's 'continuous balance' electronic **potentiometers**, by replacing the conventional galvanometer and cyclic balancing mechanisms with a simple self-contained amplifier assembly, provide continuous balance with high sensitivity and simplified and rugged construction. The models exhibited included a circular chart recorder controller with three-term pneumatic control, a multipoint precision indicator with integrally-mounted thermocouple switches, single and multipoint strip chart recorders (2 sec. and 5 sec.), a circular scale indicating electric proportional controller with manual reset, and recorders for liquid steel temperatures and radiation pyrometers. All these instruments were demonstrated, as was the **Protectoglo combustion safety systems**, operating on the principle of flame conductivity, which provide instantaneous shut-down on flame failure. The standard 'cut-off' and the integral purge timer models were also shown.

George Kent Ltd. The chemical section of the stand included a diagrammatic layout of the **instrumentation of a battery-acid plant** now in operation in this country. Instruments shown in conjunction with this layout included a **specific gravity recorder** which uses a ring balance meter

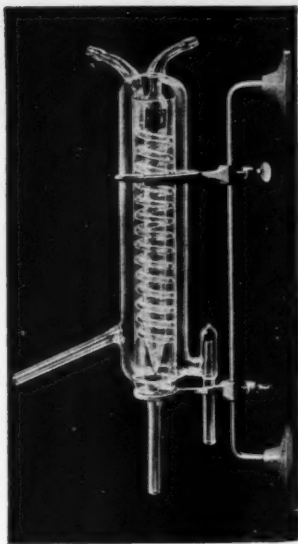


[Photo: W. G. Pye & Co. Ltd.]
pH meter.

for the measurement of the differential pressure generated through an air reaction unit by a standard and a measured liquor flowing continuously in an arrangement of comparator tubes. In this way the density of acids, etc., may be continuously recorded. For **flow measurement** the *Multepec* recorder can be used in conjunction with a rotameter. An RS/C meter for the measurement of **steam flow** was another exhibit. This is a simple flow-meter which is bolted directly into the main. Also on view was a **temperature control** arrangement utilising a mercury-in-steel temperature recorder and a Mark XX control unit. In the field of **pH measurement** Kent's showed a new glass electrode pH recorder with flow channel and immersion types of electrodes. This new instrument is claimed to be a great technical advance and is expected to eliminate many of the difficulties now associated with pH measurement.

Mullard Ltd. exhibited instruments in four groups: electronic test equipment, electro-chemical equipment, ultrasonic equipment and vibration equipment. Included in the chemical group was a new **electronic polarograph** of the type in which the current to a dropping mercury electrode is continuously measured by electronic means. Display on a cathode ray tube screen allows measurements to be made very rapidly, and an accuracy of 0.01 microamps can be achieved. Provision is made in this polarograph for the accommodation of 11 tubes of solutions to be analysed, so that, in conjunction with other design features, exact routine analysis can be carried out very rapidly. An **electronic temperature controller** has a temperature sensitive element held in close thermal contact with the water bath or other apparatus to be controlled. The resistance of the element alters with temperature and so unbalances a bridge network in which it forms one arm. The setting accuracy of the scale is $\pm 0.5^\circ\text{C}$, but once the instrument is set up it is claimed to be consistent within $\pm 0.02^\circ\text{C}$. Two other instruments which have many applications in chemical processing were the *Mullard conductivity bridge and conductivity controller* for measuring and regulating the conductivity of industrial solutions.

W. G. Pye & Co. Ltd. has recently developed an entirely new mains-operated **portable direct-reading pH meter** and commercial production has just begun. This instrument was exhibited together with a wide range of galvanometers, universal and vernier potentiometers, standard resistances, Wheatstone bridges, a cathetometer, a.f. oscillators, etc. The pH ranges in the new meter are 0 to 8 and 6 to 14, and the reading is given directly on a large meter. There are only two controls to adjust each day and, it is claimed, the readings are almost entirely unaffected by wide mains supply variations. The glass electrode is completely protected and the reference electrode is of robust construction;



(Photo: Townson & Mercer Ltd.)
'Strip-action' still

tion; automatic temperature compensation is provided. The whole instrument is completely self-contained and, weighing less than 18 lb., is readily portable. The electrode arrangement is one of the most interesting features. The essential novelty consists in surrounding the glass electrode with a carbon-filled polythene sheath, which acts as a protection and carries a temperature compensation element.

Quickfit & Quartz Ltd. displayed a wide range of **interchangeable laboratory glassware**. This included the Quickfit utility set which effectively demonstrated its versatility as 14 different assemblies can be made up from 18 pieces. New equipment included a semi-micro molecular still; spinning band fractionating still; chromatographic adsorption columns and assemblies; climbing film evaporation unit and latest designs of Soxhlet extractors. Recently introduced spherical joints were also on show.

Examples of **larger glass units** on display illustrated the progress made in all aspects of glassblowing technique and equipment design. These include a plant-scale Soxhlet extractor; a high-efficiency vacuum still, and heat exchangers. A one-twelfth scale model of a **sulphuric acid absorption plant** indicated the scope of glass units for large plant. This absorption tower is capable of producing 20 tons/week of pure sulphuric acid.

Rotameter Manufacturing Co. Ltd. The range of **flowmeters** supplied by this firm varies in size between the smallest unit for measuring 2 to 25 cc./min. of air and the largest for 40 to 400 cu. ft./min. of air. The latter instrument is of additional interest since it involves a pressure loss of less than 2 in. w.g.

The type 150 metal tube Rotameter, the type 750 low-flow alarm and the type 400 pneumatic transmitting Rotameters are instruments which operate on water flow.

Type 400 has recently been brought into production and can be used as a standard pressure gauge. The unit in this case is of all-metal construction, but models with glass tube metering elements are being produced.

A wide range of precision bore **tapered glass tubes** were also displayed, as well as large diameter precision bore, parallel glass tubes, as used for taper pole flowmeters of lead construction.

Short & Mason Ltd. The main exhibit consisted of a **graphic panel**, embodying a completely new series of transect recording and indicating receivers, which occupy a panel space of only $4\frac{1}{2} \times 5$ in., and have a built-in auto-manual control and roll type charts. These instruments were connected up to provide a working demonstration of the Transect system of pneumatic transmission for graphic panel installations. A comprehensive range of **indicating, recording and controlling instruments** for temperature, pressure, flow, liquid level, humidity, specific gravity, etc., were also shown, including nearly all possible variations of the Fulscope control mechanism. A section of this stand was devoted to standard laboratory thermometers, portable recording instruments (barographs, thermographs, hygrographs), anemometers and watch, pocket and surveying aneroid barometers.

Simmonds Aerocessories Ltd. exhibited many types of industrial and marine **liquid contents gauges** including examples of hydraulic, hydrostatic, direct-reading and electronic systems. A demonstration unit showed specimen gauges recording the rise and fall of liquid in a tank. Simmonds contents gauges may be fitted to include high or low level alarm bells and warning lights, or to automatically control pump operation. Practically any kind of liquid can be measured and for the more difficult applications where very low temperatures exist, where no metal parts may be in contact with the fluid or where agitators are in use, the industrial *Pacitor* electronic gauge has been developed.

Stanton Instruments Ltd. showed a complete range of **precision balances** to cover every need of research, industrial and educational laboratories, from a simple free-swinging instrument to a micro-balance with a sensitivity of 0.001 mg. Also shown were heavy duty balances, auto-loading balances, specific gravity balances and analytical and heavy duty weights, including those of Immaculate-5 stainless steel. The new Stanton **aperiodic balances** were shown for the first time. These have a new design of weight-loading attachment which gives positive finger tip control in weight-application even for the heaviest weights. The newly-developed precision limit balance which has wide applications in many fields of industrial control work was also on view.

Townson & Mercer Ltd. showed several interesting laboratory devices. A **vacuum**

oven has a cast, ribbed aluminium alloy door with plate-glass window, a heavy spun aluminium body with removable shelf supports, *Simmerstat* control up to 120°C, and a new type of metal vacuum tap. The insulated aluminium jacket embodies a built-in gauge, an internal thermometer, and two shelves 9½ × 9½ in. The self-centring door pivot on a cast swing arm is pulled in by a coarse thread bolt and knob.

The simplified **Karl Fischer apparatus** for the estimation of water utilises the reaction of water with iodine and sulphur dioxide in the presence of pyridine and methyl alcohol. The T & M model is based on a design developed by the British Drug Houses Ltd. It comprises a single reaction unit without mechanical stirring. The shape of the vessel is such that a jet of dry nitrogen is introduced near the bottom tap and acts as a very satisfactory stirrer.

The twin platinum electrodes in a single glass sheath pass through a tubulure and come nearly to the bottom of the vessel. The system, therefore, operates with a very small quantity of liquid. Two further tubulures and other accessories are included.

A '**strip action**' still producing distilled water from the steam mains is capable of dealing with 1 gal./hr. although the quantity may be reduced if the steam is very dirty. A flameproof **hotplate** for use in oil laboratories, a **constant humidity cupboard** and a **variable speed shaker** were some of the other exhibits.

G. H. Zeal Ltd. displayed examples of almost every type of **thermometer** in general use today. The range exhibited included a metal-cased thermometer for general industrial use, thermometers for oil refineries and glass-sheathed insulated thermometers.

follows closely the general arrangement of earlier editions. It is surprising therefore that in Fig. 43 the graph of the ratio

$$\frac{V \text{ (average)}}{u \text{ (max.)}}$$

versus Reynolds number is credited to McAdams' 'Heat Transfer' without any mention of Stanton's original work in 1914, which forms the basis of the graph. Incidentally, one of the ordinates is incorrectly labelled. Under pipe work, some information on tantalum and columbium piping is given for the first time, although the opportunities for using it must be very limited. **Size enlargement.** This title is very broadly interpreted and covers such diverse topics as extrusion of metals and plastics, moulding of plastics, powder metallurgy and flocculation. If size enlargement is intended as a corollary to size reduction then flocculation and sedimentation alone should be dealt with, plus perhaps agglomeration by supersonic vibrations and electrostatic fields. However, the odd topics are welcome, if only because they cannot find a place elsewhere. **Dialysis.** A useful addition; in three pages a brief account of theory and practice is given, together with an illustrative dialyser design calculation.

Viewed as a whole, there is a tremendous amount of useful information well set out and readily accessible for readers with some knowledge of the subject. It is unfortunate that the very completeness of the work tends to mislead students into thinking that 'Perry' is an omnibus textbook which will fill their every need. It is not a textbook and does not set out to be one. There are also some minor factors which detract slightly from its value to British readers, e.g. the list of properties of constructional materials and the reference list of engineering and industrial standards refer exclusively to American products and practice.

The quality and usefulness of the contents of the book must be a source of pride to the editor-in-chief and his board of associates. The price is not unreasonable for the type of book, but one hopes a new edition will not appear too quickly to make this one obsolete.

H. W. THORP, B.Sc., M.I.CHEM.E.

Chemical Engineers' Handbook

THE publishers made a bold but wise decision in producing the third edition of 'Perry' in a new and larger format.* Editions 1 and 2 were extremely bulky and the thin paper used for the 3,000-odd pages of the last edition was rather fragile and transparent, and the pages became 'dog-eared' by frequent use. The third edition has a page size of 9½ × 7 in., is printed in double column on very serviceable paper and runs to only 1,942 pages. The title 'Handbook,' however, now seems a little misleading and it would probably be more accurately described as a general reference book on chemical engineering and related topics.

Chemical engineering covers such a wide variety of subjects and has acquired such a voluminous literature that the selection of material to be included in a single volume must always be a matter of choice. When that choice is made by such a distinguished group of experienced men as comprise the editor's advisory board one can have little criticism of their selection. Many sections have been materially revised to include recent work, while the articles on size reduction, power generation, distillation, plant location, drying, movement and storage of materials, process control and high-pressure technique have been entirely rewritten. By omitting—wisely in the reviewer's opinion—from earlier editions some articles on report writing, physical, analytical and organic chemistry it has been possible to add new contributions on the general theory of diffusional operations, furnaces and kilns, size enlargement, azeotropic, extractive, molecular and multi-component distillation and dialysis. One is all the more surprised, therefore, to find that, in spite of these radical alterations, there are 13 pages of five-figure logarithm

tables out of a total of 47 pages devoted to mathematics!

One important result of the larger page and better (more opaque) paper is that charts and line diagrams are in most cases of a reasonable size and easily legible; with the half-tone illustrations one does not feel so happy. Mostly they are produced with too fine a screen, so that of the ten blocks in the book only two are worth producing and these should have been printed on art paper. A particularly bad instance is an illustration of a press for powder metallurgy which for all the information it gives might well be omitted. A better plan would have been to collect all the blocks on to one or two pages of art paper when the detail of the originals would have been revealed. The line blocks are good and clear, but it seems that the strict pruning in most sections has not been uniformly applied and one wonders why it was necessary to include a sketch of a sack trolley—a reader who is sufficiently experienced to use 'Perry' should surely recognise this common piece of industrial equipment by description alone.

A reference book which covers a wide field must have an index which is complete and sufficiently detailed so that subjects sought are easily found. By this criterion the third edition is very good and a vast improvement on its predecessors, although one or two small errors have been noticed.

So much for the general aspects of the work. On coming to the subject matter of the individual articles one finds a uniformly high standard, so that it will suffice to comment on a few topics taken at random, mostly from the new or revised articles.

Furnaces. This article has some good descriptive material useful for operators, but insufficient for design purposes. The scope is comprehensive, but not all would agree with the classification. **Fluid flow.** This section has been but little revised and

**Chemical Engineers' Handbook*, J. R. Perry, Editor-in-Chief. 3rd Edition. McGraw Hill, London, 1950. Pp. 1942, illustrated, £5 7s. 6d

Tin Research in New Laboratories

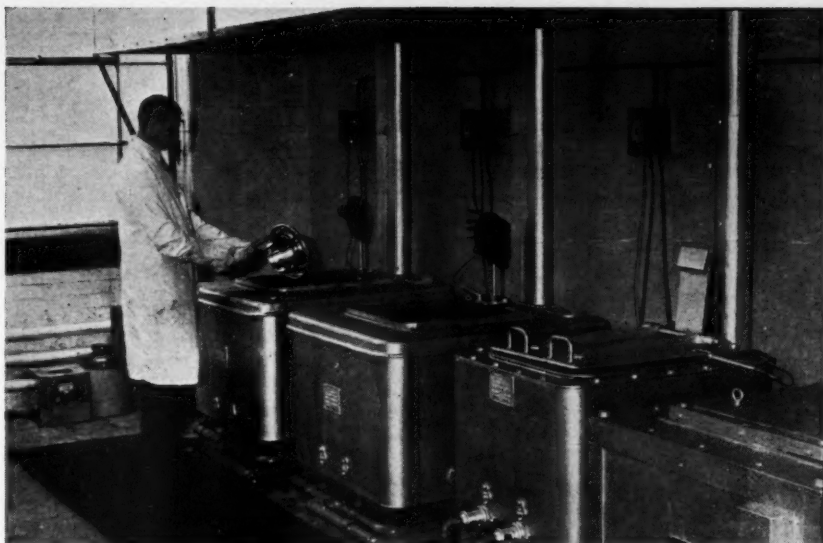
THE Tin Research Institute is a research association financed by tin producers and controlled by the International Tin Research and Development Council, founded in 1932, which consists of delegates appointed to represent the tin mining industries of the Belgian Congo, Bolivia, Indo-China, Indonesia, Malaya and Nigeria. It is the only example of international cooperation of its kind in the world.

Before 1938, the Council carried out extra-mural research. It was then decided to erect a laboratory at Fraser Road, Greenford, Middlesex, which was opened in December, 1938. By the outbreak of the war much of the scientific work was reaching a stage where facilities for semi-industrial scale investigations were required to prepare new or improved processes for industrial applications. It was then decided to build at Greenford newer and larger laboratories and to open offices for technical service overseas. A few weeks ago these laboratories were officially opened by the Duke of Gloucester.

The annual world output of tin is about 166,000 tons at the present and has a value of approximately £200,000,000. About 40% of this output is produced by the British Commonwealth. There is no tin mining industry in the U.S.A. which imports about 60,000 tons per annum.

Electrochemical section

The manufacture of tinplate for canned foodstuffs absorbs 36% of the total tin output, being the largest consuming industry. Thus, quite a large area of the new laboratories is devoted to electrochemistry and electroplating investigations. In the electrochemical research laboratory, preliminary and exploratory work is done which may ultimately lead to practical processes of tin and tin-alloy plating. The equipment includes a Hull cell, devised so that metal may



Full-scale tinning plant. Left to right: Two tanks for tinning steel and cast iron, a palm oil pot for draining off excess tin and a tin pot for non-ferrous metals.

be deposited at various current densities throughout the length of a single specimen in one operation, a pioneer tin-nickel plating bath which has been working continuously for 18 months, an alkaline sodium stannate bath and a range of electrical measuring instruments.

In the adjacent plating workshop, full-sized industrial plant is installed. Three methods of tin plating are in use, the sodium stannate bath, the stannous sulphate bath and the stannous fluoborate bath. Three new plating processes have been invented in these laboratories and are claimed to be important contributions to the science of electrochemistry. In tin-zinc alloy plating the deposit contains about 78% tin and 22% zinc. It is used to protect steel against corrosion, and for

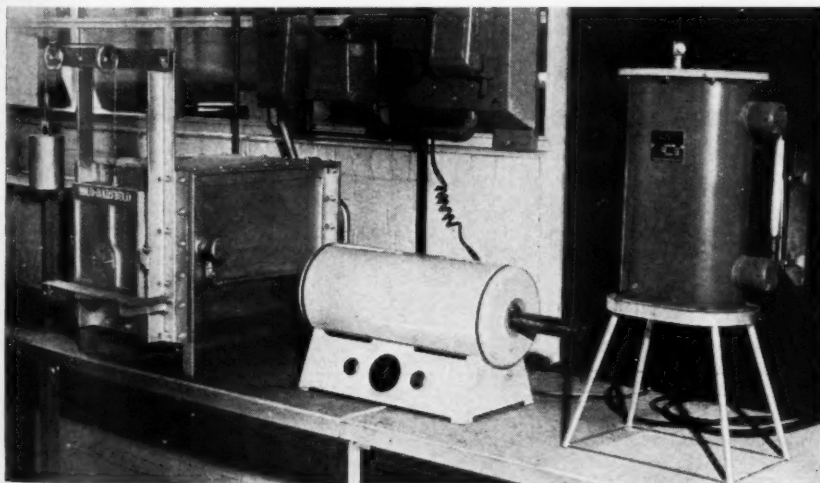
many purposes provides better protection than cadmium or zinc. It is especially valuable for protection against tropical and marine atmospheres. The second process is speculum plating in which the deposit contains about 40% tin and 60% copper. It is a decorative finish with a white colour which is said to retain its lustre indoors. These two processes are both being used commercially in several countries. The third process has not been established commercially but trial installations are being made in several countries. This method employs about 65% tin and 35% nickel. It does not tarnish indoors or outdoors and requires no cleaning. The coating has a faint rose-pink tint. Because it uses so little nickel, the process is expected to help the motor car, bicycle and similar industries badly hit by the shortage of nickel and chromium.

Heat treatment laboratory

The heat treatment section is equipped with electric furnaces which can be controlled automatically so as to maintain specified temperatures within very fine limits and for prolonged periods. Special atmospheres can be used as required.

Corrosion laboratories

The most important applications of tin and its alloys arise from their high corrosion resistance which has to be tested under a wide range of conditions. The application of a thin coating of tin (0.00005 in.) to steel before painting greatly lengthens the life of the painted steel and investigations are being made into the relative performance of tinned and untinned steel. Other work is being done on



A range of electric furnaces in the heat-treatment laboratory.

(Concluded on page 338)

Gas Turbines in the Chemical Industry

WHEN used as a power producer, the gas turbine must be compared with the diesel engine in the case of low powers or the steam turbine for high power. In comparison with the diesel engine for power, say in the region of 1,000 kW, the plant efficiency of the diesel engine is up to 50% higher than that of the gas turbine. However, the differential in fuel cost may vary between 20 and 50%, and if this is taken into account with the very much lower lubricating oil requirement, the gas turbine will on a basis of running cost be not far behind. Also to be taken into account is the gas turbine's potentially low maintenance cost and its practical independence of cooling water.

In applications where fuel cost is relatively unimportant, *e.g.* oilfield operation and units for military purposes, the low first cost of the gas turbine may make it a more attractive proposition than the diesel unit. The light weight of the gas turbine facilitates transport by air to inaccessible locations.

For high powers the coal-burning steam turbine can show a thermal efficiency about equal to that of the gas turbine. Here again the differential between the cost of coal and fuel oil proves the deciding factor. Thus on the score of fuel economy, present-day steam sets will continue to have an advantage over the gas turbine. The gas turbine has, however, some advantages over the steam set which may influence the choice in special circumstances, *e.g.* (a) a gas turbine station is lower in capital cost than a steam station, (b) a gas turbine has very low standby losses and sets can be put on load quite rapidly, (c) operating staff of a gas turbine station is smaller than that for a steam station, (d) potentially lower maintenance charges are possible with the open cycle gas turbine and (e) the gas turbine installation space is smaller than that for the steam turbine. In other words, even with high power, the gas turbine should be given special consideration for peak load sets, for standby sets and for stations with very low load factor.

Gas turbine with a waste heat boiler

In an arrangement combining a gas turbine with a waste-heat boiler, virtual independence of steam and power outputs is obtained by fitting the waste-heat boiler with an auxiliary burner. Alternatively, or in addition to this suggestion, a throttle valve may be placed between the turbine and the boiler. Without the use of these refinements, it will be appreciated that the steam generated falls with decreasing power output. To give some idea of the order of magnitude of the steam and power relationships, the following figures from an American source are given:

At design point, 3,500 kW., 80°F. ambient temp.

Turbine exhaust temp. is 820°F.

Some possible applications of the gas turbine engine in the chemical industry were recently discussed by I. Lubbock at a meeting of the Chemical Engineering Group of the Society of Chemical Industry. Among the arrangements cited were a gas turbine for waste heat recovery, combined with a diesel engine or steam turbine, and with a blast furnace. Details are summarised below.

For 75% boiler effectiveness, the rate of generation of saturated steam is 200 lb./sq.in. gauge, 26,000 lb./hr. 50 lb./sq.in. gauge, 31,000 lb./hr.

An interesting point to chemical engineers is that this steam could be used in existing turbines which might be outdated for continuous service but could be run for short periods. This would mean additional electrical power without additional steam turbine investment costs. Similarly, there are many old power plants in which boilers are being de-rated. With minor alterations, these could be converted to waste-heat boilers for gas turbines, thus saving the investment cost of the boiler.

Houdry plant

In the petroleum industry the use of gas turbines for compressing air by utilising waste heat as in the Houdry plant is too well known to be amplified here, except perhaps to draw attention to the very satisfactory service that more than 30 such gas turbines have given.

Velox boiler

Perhaps one of the most well-known methods of supercharging a steam boiler is the principle used by Brown Boveri in the Velox boiler design. This design results in a very compact furnace and also allows for considerable space savings in the boiler itself. As is well known, many of these sets have been constructed and run on a residual fuel oil.

Compounded with the diesel engine

Unlike the reciprocating engine, the gas turbine requires large quantities of gas at moderate temperature and, due to this fact, compounding the two engines can be arranged with advantage to overall economy. One example is a free-piston gas-generator-cum-gas-turbine, with the turbine only providing the power output. Alternatively, both components may combine in meeting the load, in which case the gas turbine supercharges the reciprocating engine.

Compounded with the steam turbine

A combination of steam and gas turbines

shows great promise as an efficient power unit. With comparable speeds and continuous output characteristics, matching on the one shaft is easily arranged. In this connection, reference may be made to the Mercier Equipressure steam-air plant. Both turbines are coupled to the load and the pressure of each fluid in the boiler is the same, relieving tube stresses in the high-temperature zone. Air compression is achieved by the use of specially designed free-piston generators.

Gas producers

The possibilities of the gas turbine for waste-heat recovery and auxiliary power generation in the coal-carbonisation industries are now being exploited. In one such arrangement ordered by the West Midlands Gas Board, waste gas from the retorts passes to the main air heater, where the gas is cooled from 800 to 475°C. Subsequently, the waste gas passes through an ordinary waste-heat boiler plant where it generates steam at 120 lb./sq.in., this steam being used for process purposes—chiefly for steaming the coke in the vertical retorts. In the closed cycle the air leaves the main heater at 600°C. for entry to the gas turbine which provides power for any purpose.

Blast furnace application

The gas turbine cycle is specially suitable for blast furnace use, because the air requirements of the furnace can be supplied from the compressor of the gas turbine set, thereby eliminating costly steam boilers and condensing equipment. Although the compression ratio of the compressor unit is determined by that required for the gas turbine cycle, the furnace air may be obtained at the desired pressure by bleeding off at an intermediate stage. Coupled to the turbine shaft through gearing is a gas compressor used for compressing some of the blast furnace gas which is burnt in the gas turbine combustion chamber with the preheated high-pressure air from the compressor. As the power requirements of a blast furnace are usually of the order of 10,000 kW, the gas turbine would seem to find here a favourable application.

Methane in mine upcast air

Methane occurs in the upcast from collieries in concentrations up to about 1%. Since this is of the right order for a gas turbine, it is possible to liberate this heat in a gas turbine and provide all the power our collieries require. To get such a mixture to burn, however, a considerable amount of preheating is necessary. If the methane is used in concentrations greater than about 1%, dilution with fresh air will be applied. It is more likely, however, to be used where concentrations fall below 1%, when an auxiliary fuel will need to be burned.

New Plant and Equipment

Tube diaphragm pump

A tube diaphragm pump constructed of chemically inert components is being marketed by Merrill Pumps Ltd. It is a glandless, self-priming pump specially designed for handling chemicals. Operation requires an enclosed hydraulic system connected to a piston pump and power input from any convenient source. The tube diaphragm is subjected to a small differential pressure between the hydraulic fluid outside the diaphragm and the liquid handled inside the diaphragm, but the stresses are spread uniformly over a very large area.

The pump is located and sealed inside a rigid cast iron pulsation jacket by flanged cast iron cages. The jacket is connected by a standard 2 in. flange, either directly or remotely, to a piston pump. A liquid sealed cast iron piston moves in a cast iron cylinder and by displacing perfectly clean hydraulic fluid backwards and forwards, transmits positive suction and pressure strokes to the tube diaphragm pump. Liquid sealing is accomplished by connecting a groove in the cylinder wall to a small reservoir of hydraulic fluid. The piston pump is thus not dependent on fine clearances, rings or packings.

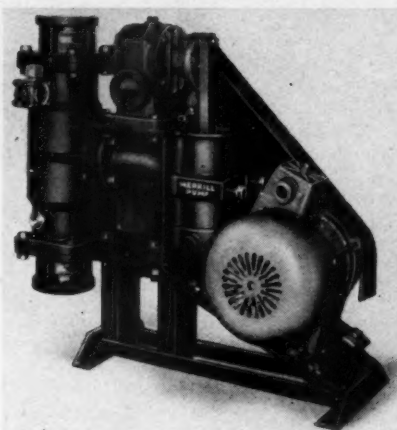
The hydraulic fluid may be water, but is preferably a mixture of water, ethylene glycol, sulphonated castor oil and inhibitor.

The tube diaphragm pump can be built into the actual pumping line and remotely operated by the piston pump. One piston pump can selectively operate any one of a number of tube diaphragm pumps. It can also operate two or more pumps at the same time, in which case the displacement of the piston pump is shared over the whole number of diaphragm pumps.

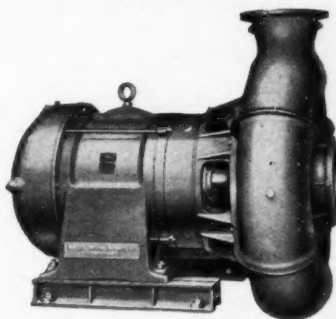
In general the pump has a capacity which can be varied from 1 to 12 gal. min. by altering the stroke of the piston. It may be operated up to speeds of 150 r.p.m. A 25-gal/min. capacity model and another 75 gal/min. pump set are available, having twin tube diaphragm pumping units.

Photoelectric drop counter

A photoelectric drop counter, using a vacuum photocell as a light-sensitive device, has been developed to facilitate the counting of drops from the microburette and similar devices by the Chemical Defence Experimental Establishment. It operates in the drop size range from several mm. to 0.25 mm. diameter and at count rates up to 400/min. The passage of each drop through a light beam produces an electrical impulse at the photocell. These impulses are amplified, discriminated, and then utilised to initiate a 'flip-flop' circuit controlling a mechanical register. Provision is also made for continuous monitoring by a cathode-ray oscilloscope. Stabil-



Tube diaphragm pump constructed of chemically inert components.



Stainless steel pump by Rhodes, Brydon & Youatt Ltd., believed to be the largest of its kind. Details were given in *INTERNATIONAL CHEMICAL ENGINEERING*, June, p. 287.

ised power supplies for the photocell and amplifier circuits are drawn from a built-in power pack operating from 200-250 V, 50 cycle, AC mains.

Heating jackets

An efficient means of uniformly heating laboratory and manufacturing glassware is provided by *Stabilag* jacketing, which is constructed from *Fibreglass* cloth, to which is stitched a closely pitched resistance wire, lying between two further layers of *Fibreglass*. The heat dissipation of the jacket is such that the whole jacket surface rises to the operating temperature almost instantaneously when switched on. The jacket is suitable for either single- or three-phase A.C. or for D.C. With its double thickness fabric, pre-compressed lagging and rigid metal shell, the jacket can support the load of a vessel full of liquid. It is not confined to indoor use; mild steel, stainless steel or copper can be used in place of the normal aluminium shell for outdoor operations. A great advantage is that the jackets are complete units and do not require special supports.

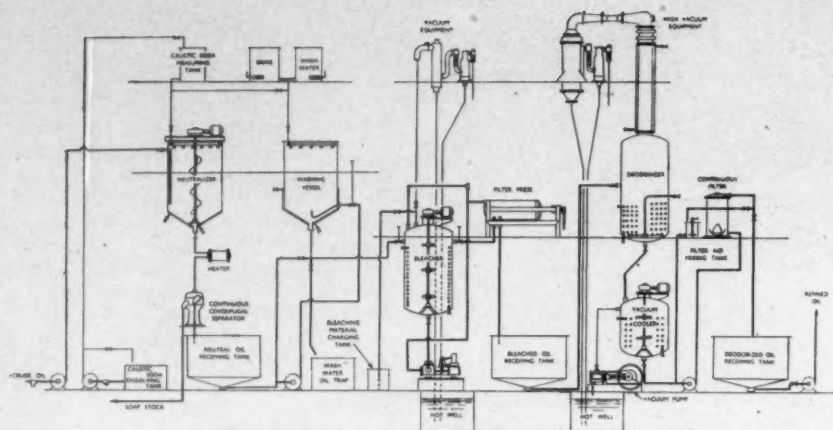
The danger of local over-heating in the jacket, following a falling of the level of the liquid in the vessel as evaporation proceeds, has long been a stumbling block to the general acceptance of this form of heating. This danger has been eliminated in the *Stabilag* jacket. The construction is such that, should a jacket be left on and the vessel becomes empty, the temperature will not rise above 550°C. The glass fabric will comfortably withstand this temperature. The danger of shorting and burn-out due to accidental spilling of conducting fluids on to the jacket surface is reduced to a minimum by the method of winding the heating element.

Many solutions must be filtered hot, and close control of the temperature is often highly important, for if there is too much heat low-boiling solvents will boil away, yet for some purposes a higher temperature is required than can be furnished by the usual means of heating filter funnels. The *Stabilag* funnel jacket will overcome these difficulties. By means of a sensitive element attached to the jacket accurate control to within $\pm 0.1^\circ\text{C}$. is possible. The element has a variable gap across the precision contacts and the extremely small current (0.00005 A) actuates a relay without danger of 'arcing,' hence risk of damage to the control surfaces, with resulting breakdown, is avoided. The amplifier is so designed that if a process involves continuous operation with unattended periods, it can be fitted with a unit which will cut the current supply at a preselected temperature; it can easily be reset.

The *Stabilag* Co. are manufacturers of the jackets.

Vegetable oil refining plant

The refining of vegetable oils may be subdivided into the following principal unit operations: (1) neutralising, preceded by degumming or separation of lecithin where necessary, (2) washing, (3) drying, (4) bleaching, and (5) deodorisation. There is no sharp division between one unit operation and the other and the plant is designed in an integral manner for maximum economy and ease of operation. The Power-Gas Corporation Ltd. produce refining plant capable of handling all vegetable oils such as groundnut, cottonseed, linseed, palm and coconut oils, etc., as well as animal fats such as fish and whale oils. In choosing the correct method of neutralising, the following factors must be borne in mind, namely, the type of oil to be processed, the free fatty acid content of the oil, and the desired plant capacity. Power-Gas supply plants utilising the cold neutralisation process (which is based on a number of fundamentally new concepts), the gravity settling process, and the short-mix process. After neutralisation the washed oil is transferred by gravity or suction into vacuum drying and bleaching vessels, where it is heated to the requisite temperature and dried under an absolute



Flow diagram of edible oil refining plant.

pressure of 2 to 3 in. Hg. The oil is then treated with bleaching earth and a small quantity of activated carbon, drawn into the vessel by vacuum. A high-speed agitator intimately mixes the oil and bleaching materials. The treated charge is pumped through a filter press to the bleached oil receiving tank. Four charges are processed in this manner in 24 hours. The bleached oil is drawn into the deodoriser by vacuum and heated to the requisite temperature which depends upon the oil being treated but is of the order of 200°C.

Air heaters and steam removers

A new development in heat transmission equipment is a miniature fan heater, the *Spiralux* wall mounting steam model, with all-copper heating element, suitable for steam pressures up to 150 lb./sq. in., and with an output of 7,000 to 10,000 B.Th.U./hr. accordingly. This steam model is particularly advantageous for space heating where an existing steam supply can be utilised, and is also a useful standby in these days of load shedding. An all-electric *Spiralux* fan heater is also available incorporating automatic thermal cut-out and built-in switch and rated 2 kW. and 3 kW.

A similar device is the steam removal unit for laundries and dyeworks. This system, which incorporates a heating unit and an exhaust fan, is claimed to have proved very successful in eliminating excess moisture from the atmosphere.

The fan heaters and steam removal unit are supplied by Spiral Tube & Components Co. Ltd.

Sheet catalyst

According to a recent U.S. patent, a solid catalyst in the form of sheets, screens, grids, etc., used instead of the usual granules, improves temperature and reaction control in catalytic units of the stationary bed type as employed in hydrocarbon conversions.

The catalyst member may be any of several compressed forms which can be inserted in the tube. It can be folded, a

New Plant and Equipment
Fill in the Enquiry Coupon on page 342 for further details of the equipment and plant described in
INTERNATIONAL CHEMICAL ENGINEERING.

coiled volute or flat spring plate, or a wire shaped into a helical spring.

The main advantage held by a solid catalyst over the granular catalyst is said to be better heat control. It comprises a folded screen, coated with catalyst which is held in proper shape by metal supports, consisting of crossbars in the form of a simple Greek cross. Additional supports may be used between the ends of such an assembly, and a longitudinal support bar may also be included to provide rigidity in the screens. The screen is of fine wire mesh, and the coating may completely fill and cover the meshes or cover only the individual wires, maintaining the open mesh to allow better flow of reactants.

Because there is less resistance to reactants flow, catalyst in this form provides for low-pressure drop. The catalyst is easily removed and replaced, moving out of the tube as a unit, and there is no fusion of small particles to the tube walls.

Electrostatic precipitator

In our April 1950 issue (p. 181) we gave details of a high-voltage rectifier for the electrostatic precipitation of noxious gases installed in the chemical works of Vieille-Montagne, Belgium. This information was supplied by the makers of the equipment, Partridge, Wilson & Co. Ltd. This firm now inform us that their customers feel that the note implies that previously they had no system to treat the gases before discharge to the atmosphere. In fact, the new precipitator replaced a Lurgi gas treatment system.

Lightweight industrial gloves

A new lightweight commercial glove made of PVC has been introduced by James North & Sons Ltd. These gloves

are intended primarily for abrasion resistance in handling materials of all kinds and are lighter and more supple than the standard weight of PVC gloves. They are said to effectively replace the usual cotton gloves, cotton-backed leather palm gloves and lightweight leather gloves. Trials have recently been carried out by a number of users to test the comparative durability of the PVC gloves against that of the types mentioned. It is claimed on the basis of these tests that the working life of the former is 15 times greater. These gloves are made with knitted wrists and are available in men's and women's sizes.

Tin Research Institute

(Concluded from page 335)

specimens of tinplate treated with *Protectatin*, a process developed at the Institute, which makes tinplate more resistant to staining when in contact with sulphur-containing foods. Accelerated and tropicalisation corrosion tests are also carried out under various conditions.

Analytical laboratory

In the analytical section, rapid and accurate analysis can be made of all the metals, alloys and chemicals used in the Institute, and few research projects could be brought to a successful conclusion without frequent recourse to this service. The equipment includes a vacuum distillation unit for the determination of certain more volatile alloy constituents in tin which has a high boiling point. Solvent extraction is also used for the detection and separation of the constituents of an alloy. Polarographic analysis apparatus is available for detecting and measuring very small quantities of metals in solutions as well as a photoelectric absorptiometer.

Other facilities

In addition to these laboratories there are various other sections including a polishing room equipped with conventional machinery. There is a hot-tinning workshop which contains full-size equipment for industrial scale investigations. In the tinplate laboratory research is carried out to improve the manufacturing process and the quality of tinplate. In this section one unit which was designed and built at the Institute, simulates conditions in modern high-speed electro-tinplate manufacture. It is used for studying the effect of the quality of the steel, methods of preparing the steel and plating conditions on the quality of the electro-tinplate. A microscope room, a mechanical testing laboratory, a bearing testing laboratory and the foundry and general metallurgical workshop complete the facilities. In the latter section three furnaces for melting metals can be attached to a high-frequency generator. These are induction furnaces which are especially useful as they reduce the risk of contamination by products of combustion.

World News

GREAT BRITAIN

Brotherton's new plants

Large new plant extensions for the manufacture of hydrosulphite, erected by Brotherton & Co. Ltd., are lying idle for lack of sulphur. Giving this news to shareholders at the annual meeting, the chairman of Brotherton & Co. Ltd., Mr. B. L. Ratcliffe, said that in view of the essential nature of the firm's products he hoped they would receive a reasonable share of future sulphur supplies. However, owing to the uncertainty of this, he could not accurately forecast the future. If supplies did not fall below their present level satisfactory results could be expected, however.

Generally speaking, 1950 had been a most satisfactory year, with a high demand for hydrosulphite, sulphides and by-sulphites. Output of sulphuric acid and sulphate of ammonia increased still further to a record figure. The firm uses gasworks' spent oxide exclusively for the manufacture of these products and because of this continued full manufacture in the coming year was expected. Increased activity of the plastics industry called for a higher output of hexamine and, to meet this, further extensions of plant capacity were in hand.

During the year the firm's tar distillery at Litherland worked to capacity. Early in the year, as the result of the integration of several gas undertakings in the north-western area under the North-Western Gas Board, negotiations were commenced which resulted in the Litherland works being merged into the co-operative scheme of Lancashire Tar Distillers Ltd. as from January 1, 1951. The full settlement under this merger has not yet been completed.

New Quickfit agents

Quickfit & Quartz Ltd. have arranged for J. W. Towers & Co. Ltd. to distribute their laboratory ware. This arrangement does not include Quickfit & Quartz's chemical or industrial glassware, nor will Towers be sole distributing agents for the firm's laboratory ware.

Pump makers acquired

The Pulsometer Engineering Co. Ltd., pumping, refrigerating and filtration plant manufacturers of Reading, have acquired the total share capital of Joseph Evans & Sons, of Wolverhampton, who have been pump makers since 1810. Each organization will continue to operate separately.

Licensing system for magnesium

Because of increasing defence needs an Order introducing a licensing system for the distribution and use of virgin magnesium and virgin magnesium alloys has been made by the Minister of Supply. It provides that any person proposing to acquire, treat, use or consume any magnesium of this kind must have a licence

and it became effective on June 11. Steps have been taken to increase supplies, including long-term contracts with overseas suppliers and the reopening of U.K. magnesium plants which were in operation during the war. First production from these reopened plants will commence this month and will be expanded as quickly as possible to meet the rising demand.

Chemistry at the British Association meeting

The annual meeting of the British Association for the Advancement of Science will be held at Edinburgh from August 8-15. The President, The Duke of Edinburgh, will deliver his address, 'The British Contribution to Science and Technology in the past Hundred Years,' on the evening of August 8. Other papers and discussions of interest to chemical engineers and chemists include 'The Changing Face of the Chemical Industry,' 'Organic Chemistry in the Service of Mankind,' 'Metals and Alloys,' 'Technical Education in the United States,' 'Chemistry at the Mid-century' and 'Hormones of the Pituitary Gland.'

A full programme giving detailed arrangements will be available in July from the Secretary, British Association, Burlington House, Piccadilly, London, W.1.

Institute of Physics officers

At the recent annual general meeting of the Institute of Physics, Prof. W. E. Curtis, University of Durham, was re-elected president. Dr. J. Topping was elected a vice-president and Mr. O. W. Humphreys and Dr. S. H. Piper were elected ordinary members of the board. The honorary treasurer, E. R. Davies, and the honorary secretary, Dr. B. P. Dudding, were re-elected.

Tyrer's managing director retires

Mr. W. Turcan, for the past nine years managing director of Thomas Tyrer & Co. Ltd., retired on May 31. He is succeeded by Mr. A. W. R. Chandler and Dr. J. S. Pedder, who become joint managing directors.

Course on radiation equipment

Physicists and engineers who wish to obtain specialised knowledge of electronic instruments used in nuclear physics, radio chemistry and work with radio isotopes are being offered a course at the Atomic Energy Research Establishment, Harwell. This is the second time that Harwell has offered facilities to specialists from industry and outside organisations (see INTERNATIONAL CHEMICAL ENGINEERING, April 1951, p. 155).

The course will last for one week, starting on July 23 and ending on July 27, and will deal specifically with the use of electronic equipment for detection and measurement of radiation. The number

that can be accepted on the course is limited to 12, but if there is sufficient demand, further courses will be arranged. Applications should be made to the Electronics Division, A.E.R.E., Harwell, Didcot, Berks.

Carbon black output to be doubled

A new unit which will double present capacity to a total of 40,000,000 lb. p.a. is to be added to the carbon black factory of Cabot Carbon Ltd. at Ellesmere Port, Ches. This factory, which was fully described in INTERNATIONAL CHEMICAL ENGINEERING, August 1950, began production last July. The firm is wholly owned by the U.S. carbon black manufacturers, Godfrey L. Cabot Inc. The new unit will produce primarily high-abrasion furnace grades of carbon blacks best suited to the full reinforcement of rubber, from liquid hydrocarbons such as petroleum refinery residues.

Contractors for the expansion will be the Lummus Co., London. Construction will probably require at least a year, depending on availability of steel and other critical equipment. Any additional financing required will be provided by Godfrey L. Cabot Inc.

Course on adhesives

Lectures, practical work and demonstrations on the technology of synthetic resin adhesives will be given at a course arranged by the Aero Research Ltd. at St. Catherine's College, Cambridge, from September 23-29.

ITALY

Mercury production up

Italian production of mercury ore in the first quarter of 1951 amounted to 48,377 tons, against 34,594 tons in the corresponding period of 1950. Due to the favourable situation on foreign markets, existing mines have stepped up their output and some mines, abandoned for years, have been reopened. Output from the latter will increase progressively during the next few months.

The price of mercury on the Italian market is now stabilised at 130,000 to 140,000 lire per container of 34.5 kg.

Petroleum chemicals plant to be built

The Montecatini Co. are to build a plant for the production of olefines and their end-products, with the help of about 2,700,000 Marshall Plan dollars, according to the E.C.A. The plant will be located at Ferrara, which is close to Italy's north-eastern coast. It will be the second plant of its type in Western Europe; the only existing plant is located in England. The Montecatini plant's capacity will be about 10,000 metric tons p.a. of ethylene, about the same amount of propylene, and minor amounts of butylene and liquid and gaseous by-products. Among the end-products are acetone, butadiene and styrene monomer, polythene, and diacetone alcohol.

BELGIUM

Oil refineries erected

A topping column, part of the expansion being undertaken by the Raffinerie Belge des Pétroles, has been erected. The tower is 30 m. high, weighs 38 tons and is 2.20 m. diam. It is designed to handle 1,200,000 litres/day of crude products. One of the refinery's two new storage tanks is now completed. Of 52 m. diam., these tanks will each have a capacity of 23,000 cu. m. of crude oil, the equivalent of a full cargo of one of the larger tankers. When completed, the new installations will enable the refinery to handle approximately 300,000 cu. m. of crude oil annually.

The new installations of the Société Industrielle des Pétroles on the Kruischans side of the new petroleum dock should be completed very soon. Erection of the Esso Standard refinery on the opposite side of the dock will not begin before next year.

SWITZERLAND

Sugar refinery project

Swiss agricultural organisations are pressing the Government to set up a second sugar refinery. The only other refinery at Arberg, Berne, has reached the limit of its processing capacity.

The organisations have prepared plans for a factory to be built at Andelfingen, Zurich. It would have an average processing capacity of 10,000 to 12,000 10-ton wagon loads of sugar beet p.a., and would work as an affiliate of the old refinery. Construction costs were estimated at about 24,000,000 francs. The new factory would increase domestic supplies of sugar from 24,000 tons to 36,000 tons p.a., leaving 134,000 tons to be covered by imports.

Although the Swiss authorities are reported to be in favour of the plans, no concrete steps have yet been taken. Swiss industrial and trade circles are still objecting to the plan which, they claim, will increase the price of sugar.

Record chemical exports

Swiss exports of chemicals and pharmaceuticals in the first four months of this year reached all-time record figures. Exports of chemicals, excluding paints and lacquers, increased to a total value of 251,900,000 francs from 155,700,000 francs in January-April, 1950. Exports of pharmaceutical preparations rose to 94,400,000 francs in January-April 1951 from 65,400,000 in the corresponding period of 1950. In the same periods exports of aniline dyes and indigo rose from 61,700,000 to 99,500,000 francs; industrial chemicals from 21,800,000 to 42,900,000 francs and perfumery products from 6,800,000 to 15,200,000 francs.

The most important customers for Swiss aniline dyes in the period under review included France, Italy, U.S.A., Britain, Germany, Holland, Sweden, India

and several Latin-American countries. The foremost buyers of pharmaceuticals included Turkey, Belgium, Italy, Egypt, Pakistan, Venezuela, Brazil and Iran. Swiss perfumes went mainly to France, Italy, Britain, U.S.A., Sweden, Germany and Spain.

GERMANY

New cellulose process

A new method of producing cellulose which uses straw in place of the wood is said to have been developed in the State-owned synthetic fibre works at Trebsen in the Eastern zone. It is estimated that 15,000 cu. m. of wood could be saved annually if this method is adopted. The new type of cellulose is claimed to be equal in quality to that produced from wood pulp and being tougher is especially suitable for ropes and twine.

High potash output

West Germany's production of potash fertiliser salt in the fertiliser year 1950-51 reached 986,000 tons, of which 641,000 tons were sold on the domestic market and the balance exported, according to a director of the Potash Sales Office Ltd., Hanover. He pointed out that in 1946-47, output reached only 269,000 tons, of which 31,000 tons were exported. The current rate of production is sufficient to meet all domestic requirements, even though only 39% of the pre-1939 all-German capacity is located in the Western zones.

Synthetic rubber industry revived

The chemical works at Juels, Westphalia, has now been permitted to reconstruct four butadiene furnaces partly dismantled in 1949. The furnaces are expected to produce 500 tons of synthetic rubber monthly. The works also received permission to operate six more furnaces for styrol production. Butadiene production is expected to begin in October, while the first output of buna is expected in November. The factory plans to use a new method of producing butadiene.

AUSTRALIA

Overcoming cement shortage

The shortage of cement is expected to be overcome by 1954 in all states except Victoria. This is forecast in a review of the Australian cement industry prepared by the Federal Division of National Development. Accordingly production should rise by 50% by 1953 under the present expansion schemes of most Australian cement works (see INTERNATIONAL CHEMICAL ENGINEERING, March 1951, p. 137, and May, p. 239).

The review suggests that the gypsum deposits of South Australia might be used for cement making and for producing sulphuric acid on lines similar to overseas methods. It also suggests research into the use of cement-like materials and some volcanic deposits which are used successfully in the U.S.A. to dilute cement.

Sillimanite exports

Many enquiries for sillimanite, mined at Mount Crawford by the Australian Industrial Minerals, are being received, according to the company's secretary, Mr. S. R. Hoskins. Sillimanite is a heat-resisting metal used, among other things, in linings of furnaces and boilers. It has been used in the Port Pirie smelters since 1906, but has only been available to other consumers since 1938.

The sillimanite is shipped overseas in the crude form in which it is mined. Recently Britain and France have taken substantial amounts and enquiries have been received from Sweden, Italy, Czechoslovakia and Argentina.

SOUTH AFRICA

H₂SO₄ plants converted to pyrites

Although sulphur is very scarce, users of sulphuric acid in the Transvaal have not suffered serious cuts because acid manufactured at Moddersfontein is produced entirely from iron pyrites. In an effort to counter the general shortage of sulphur, African Explosives and Chemical Industries Ltd. are planning to convert their sulphur burning plants to pyrites. Burners at Umbogintwini will be the first to be converted.

There is an acute shortage of many other industrial chemicals, and supplies in liquid form, even though available, are held up by a shortage of steel drums.

CANADA

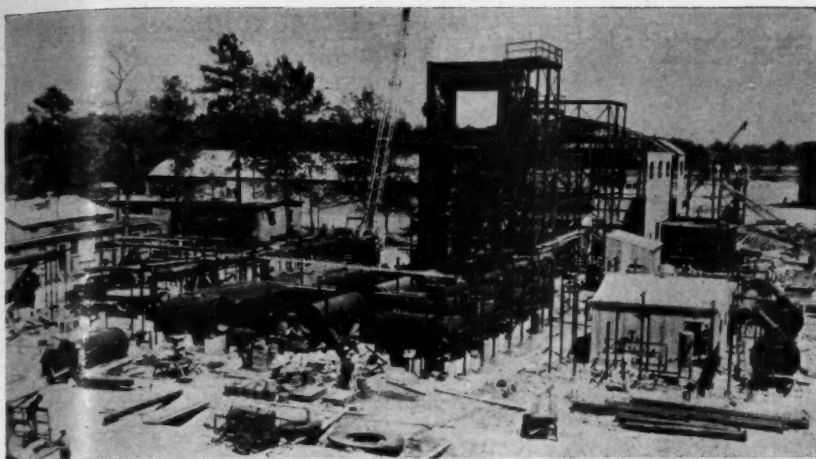
Sulphur development programme

A huge sulphur development programme in Canada may result from the decline of reserves in the U.S., according to J. R. Donald, Chemicals Director in the Defence Production Department, Ottawa. Industry will spend about \$11,000,000 in capital investment this year to explore the possibilities of increasing Canadian production of sulphur.

He estimated that by 1952 the programme should begin to take effect, with benefit to Canadian industry, employment and dollar reserves. Canada's dependence on U.S. supplies was expected to decrease and industry to cut down on imports.

The U.S. supplied Canada last year with 290,000 tons of elemental sulphur. This year, Canada needed 415,000 tons from America in addition to the 311,000 tons she expected to produce herself. Nearly all the 415,000 tons is expected to be provided by the U.S., because she is dependent on Canadian newsprint, in which sulphur is a vital ingredient. About 90% of Canadian newsprint goes to the American market.

Three sources of sulphur are available: smelter gases, iron pyrites and natural gas in Alberta. Canadian Industries Ltd. have recently announced that they will erect a new works at Copper Cliff, Ontario, to produce liquid sulphur dioxide from by-product gases of the oxygen flash-smelting



NEW CARBON BLACK PLANT TAKES SHAPE

The new \$2,000,000 Continental Oil Black Co. plant for the production of high abrasion furnace black from oil begins to take shape at Westlake, U.S.A. The new plant has an annual capacity of 25,000,000 lb. Rise in the use of synthetic rubbers, especially 'cold rubber,' has increased substantially the demand for high abrasion furnace black. Output of the new plant will be distributed by Witco Chemical Co.

process recently developed by the International Nickel Co. It is expected that the new plant will produce some 90,000 tons of liquid sulphur dioxide a year.

Liquid sulphur dioxide used at present in Canada is obtained from the U.S., but consumption is small, due to high cost. The Copper Cliff plant's output will be available to consumers at a fraction of the cost of U.S. liquid sulphur dioxide, it is stated.

Potential large consumers are sulphite pulp, newsprint and sulphuric acid manufacturers who now depend mainly on elemental sulphur. Liquid sulphur dioxide yields sulphur dioxide gas directly and tests have shown it to have decided advantages for this purpose over elemental sulphur.

Zinc concentrates contract

Another aspect of Canadian sulphur development is the news that the American Zinc, Lead & Smelting Co. has also negotiated a contract for the purchase of Canadian zinc concentrates with Barvue, a subsidiary of Golden Manitou Mines Ltd., Canada. It involves American Zinc's purchase of 175,000 short dry tons of zinc concentrates and it is understood that the deal involved about \$35,000,000.

The concentrates will be shipped to Arvida, Quebec, where the Aluminium Co. of Canada will erect a \$3,000,000 plant for the extraction of about 35,000 tons of sulphuric acid annually for its own use. The remaining zinc-carrying concentrates in solid form will then be shipped to the U.S.

The zinc concentrate is expected to run to approximately 56.4% zinc and 31% sulphur. Although no time has been scheduled for the operations to begin, it is expected that by next March Barvue will

be producing at the rate of 4,000 tons of ore daily, which should yield between 5,000 and 6,000 tons of concentrates a month. It is estimated that it will take two to three years, at an ore mining rate of 4,000 tons daily, to provide 175,000 tons of zinc concentrates. Barvue, through Golden Manitou, will spend between \$6,000,000 and \$7,000,000 for construction of a mill and otherwise preparing for underground work.

Butanediol from molasses

Sugar-beet molasses, a cheap and plentiful agricultural by-product, has been successfully converted into butanediol by a process developed in the Canadian National Research Council's Division of Applied Biology. Rapid fermentations were obtained in laboratory tests with three micro-organisms. When it was found that their yields were so close to theoretical limits that further improvements would be unlikely, pilot-plant tests were undertaken and completed, using one of the three organisms.

Since it is commercially better to use continuous rather than batch sterilisation, a continuous method for mashing, sterilising and cooling was utilised which gave satisfactory mash for fermentations. As the work proceeded, changes were made in the design of agitation and aeration systems of the pilot-plant fermenter, which resulted in better yields of butanediol. By this process 1,000 lb. of molasses yielded on the average 176.7 lb. of butanediol, 40.9 lb. of ethanol and 7.4 lb. of acetoin.

Pilot-plant data have now been used in the preparation of flow sheets and cost estimates for a full-scale industrial plant which would have an input capacity of 60,000 lb./day of molasses.

INDIA

Canadian chemical engineering scholarships offered

Under the technical co-operation scheme of the Colombo Plan, the Government of Canada has offered the Government of India 25 scholarships for postgraduate study in certain subjects, including all branches of engineering and chemical engineering. Part of the facilities offered will be in the form of travelling fellowships.

Under the scheme the Government of Australia has already offered 50 scholarships to Indian students.

Microscopes manufactured

The Government precision instrument factory in Lucknow has produced the first all-Indian microscope, it is reported. The Government hopes that the microscopes manufactured by the factory will be on sale by July. Sixty instruments a month is the present production target. Each microscope will cost Rs.550, compared with about Rs.880 for imported models.

Twenty-third cement factory being built

A new cement factory under construction for the United Provinces government is expected to be in full production by 1952. The plant, which will cost Rs.30,000,000, will be the twenty-third cement factory in the country and will have an output of 700 tons of Portland cement daily. At present, India's total annual cement production amounts to 2,700,000 tons. The output of the new plant will be used in the construction of new dams and in other projects planned by the Government.

PAKISTAN

New chemical plants

A distillery is being set up at Mardan, in Pakistan's Northwest Frontier Province, to produce industrial alcohol. It is being built by the Frontier Sugar Mills & Distillery and is expected to begin production this year. The Premier Sugar Mills at Mardan, the biggest sugar mill in Asia, has also completed plans to open a distillery in the district for processing molasses.

A factory for producing ammonium sulphate from coal for use as a fertiliser is to be set up shortly in Pakistan. Details for the procurement of machinery and materials are now being worked out by the Government. The production capacity of the proposed factory is expected to be 50,000 tons a year.

VENEZUELA

Rayon plant planned

The Celanese Corporation of America has announced the formation of a new rayon yarn manufacturer, Celanese Venezolana. Celanese Corporation has subscribed over 50% of the capital of the new concern, which plans to erect a plant near Caracas. It is expected to be in operation within about a year. Venezuelan textile interests co-operated in setting up the

company, which will be the first rayon yarn producer in Venezuela and will employ Venezuelan chemists and engineers trained in the Celanese Corporation's U.S. plants.

PHILIPPINES

First H₂SO₄ plant

The first plant to produce sulphuric acid in the Philippines is to be established soon, according to a statement by the president of the newly founded Chemical Industries Organisation. Machinery and equipment is awaiting shipment from Japan and the factory is to be set up just outside Manila. Operation is likely to start in August. Initial capacity will be 10 tons per day and, although financing is on a modest scale, it is planned to expand the project as local demand for basic chemicals increases. The bulk of the sulphuric acid produced would be used for agricultural purposes.

U.S.A.

Faster synthetic rubber process

A method of producing rubber in 8 hours instead of 12 to 14 hours without accumulating a great amount of heat in the plant has been developed by the Phillips Chemical Co. A company spokesman said that cooling coils which removed the heat more quickly, were inserted in the synthetic rubber reactors at the Borger, Texas plant. The method has been known for some time but the rapid accumulation of heat in the plant had prevented its use. It is claimed that the process will lead to a 20% increase in output with only minor plant changes and could eventually result in a 50% rise in production.

Petroleum chemicals project

A joint announcement of the formation of American Petrochemical Corporation to be owned equally by Cities Service Co and Firestone Tyre and Rubber Co. states that plans have been developed for the construction of a large modern chemical plant, possibly to be situated at Lake Charles. Its production ultimately would

encompass the fields of plastics, synthetic rubber, additives for lubricating oil, cracking catalysts, anti-freeze and high volume petroleum chemicals.

Cities Service now operates a 160,000 barrel refinery at Lake Charles, which would supply light hydrocarbons to the projected new plant. Firestone operates a Government-owned synthetic rubber plant adjacent to the refinery.

GREECE

Nitrogen plant project

The Greek Ministry of Co-ordination will shortly invite tenders for the construction of a nitrogen plant, the building of which will be financed in part by the Central Loans Committee. The Deputy Premier, commenting on the project, said: 'We are proceeding towards the materialisation of this great recovery project, which will make available a cheap fertiliser and will, together with irrigation, contribute towards a substantial increase in our agricultural production and a reduction of costs.'

TANGANYIKA

New vegetable oil refinery

The Acting Governor recently laid the foundation stone of the buildings for a new plant of Victoria Oil Mills Ltd. in Mwanza. The new factory will not only stimulate cottonseed production, but provide a welcome source of edible oils. It is being supplied by Bamag Ltd. of London and will represent the latest in cottonseed oil refining technique. It will include the new high-vacuum non-drip deodoriser, which needs less steam and cooling water than other deodorisers.

International Conferences

July 14-21. International Welding Congress, London and Oxford.

July 16-21. Conference on automatic control arranged by the Department of Scientific and Industrial Research at the College of Aeronautics, Cranfield, Beds.

Recent publications

Removal of moisture. Birlec *Lectro-dryers* can be used to exclude air-borne moisture from tanks and other storage containers and to dry combustion-air and other gaseous reagents to forestall the formation of corrosive acids. This and the many other applications of *Lectro-dryers* in the processing of food and drugs in the metallurgical industry and in conjunction with power generation and compressed air units are described in a fully illustrated brochure recently published by Birlec Ltd.

Interchangeable spherical glass joints. In the last 20 years the design of glass apparatus in the laboratory has been revolutionised by the ready availability of conical ground joints of a precise standard of interchangeability. For certain purposes, however, the advantages derived from a spherical joint outweigh the disadvantages of greater size and necessity for individual clamps. A typical example of this is at the joint between a flask and the bottom of a fractionating column in certain oil distillations. A leaflet published by Quick-fit & Quartz Ltd. describes their range of interchangeable spherical ground glass joints.

Chemical plant. The comprehensive range of chemical plant supplied by the Kestner Evaporator & Engineering Co., Ltd., is detailed in a new brochure. Acid handling and recovery plant, air and gas driers, crystallisers, distillation plant, drying plant, electric heating equipment, evaporators, fluid heat transmission systems, gas absorption units, lead linings and castings, metal pickling plant, stirrers, mixers, and sulphur burning plant are listed and illustrated.

Correspondence

Chemical Engineers in the Gas Industry

Dear Sir.—In INTERNATIONAL CHEMICAL ENGINEERING for June 1951 you give a very good summary of the Convention of the Graduates' and Students' Section of the Institution of Chemical Engineers. I wonder if you could find space in your next issue to correct a statement which is attributed to me, as follows:

'Although the industry was entirely dependent upon contractors for all its plant and there were thus no openings for the chemical engineer as a plant designer within the industry itself . . .'

What I should have said—and what I hope I did actually say—was as follows: 'Although the industry was entirely dependent upon contractors for all its plant, there were many openings for the chemical engineer as a plant designer within the industry itself and also a great demand for chemical engineers to investigate the co-ordination and balance of all processes.'

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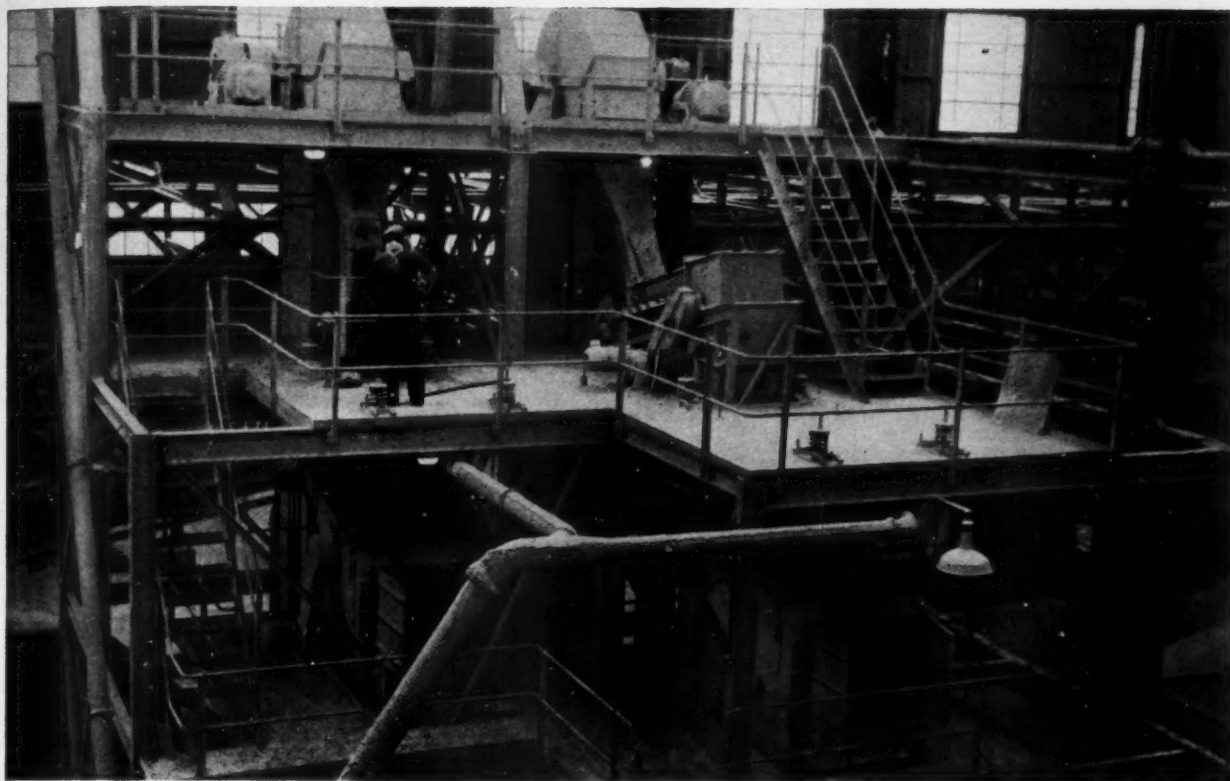
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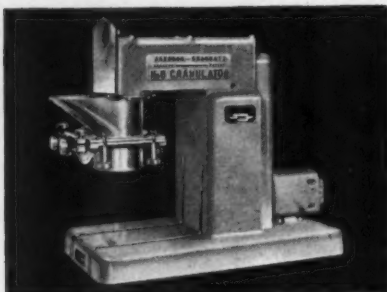
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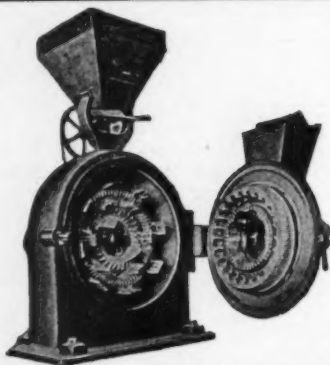
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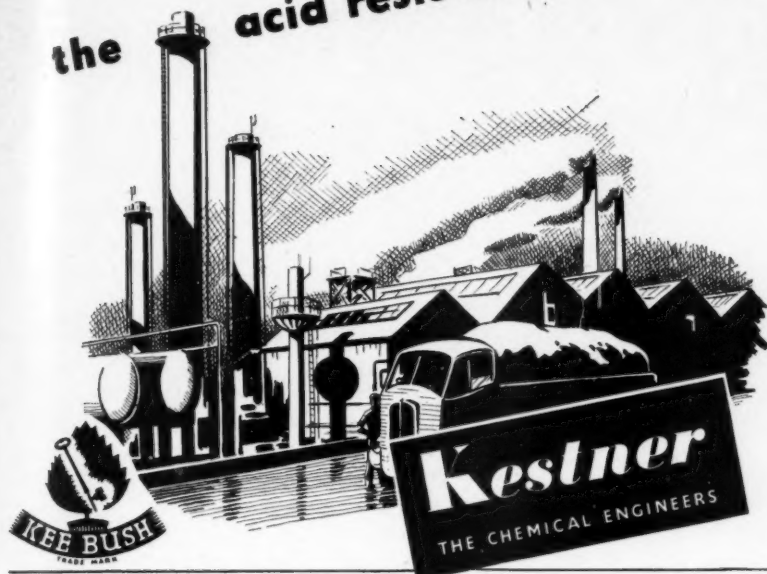
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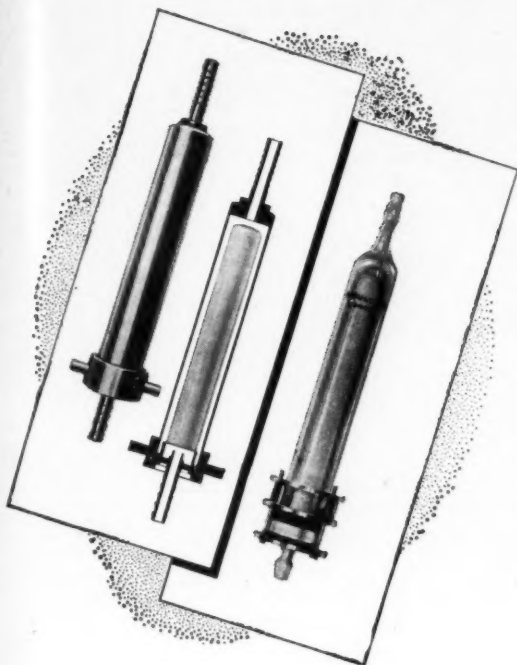
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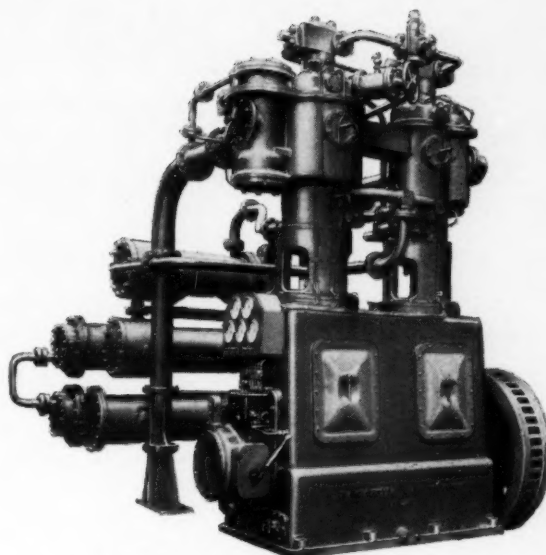
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